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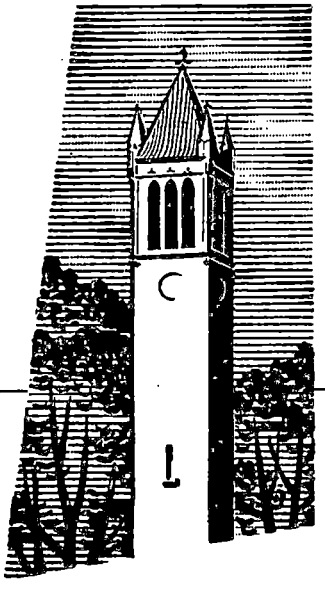
# SOIL STABILIZATION WITH LIME

by
D. T. Davidson
and
Associates





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D. T. Davidson, Professor, Civil Engineering
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**of the**
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## PREFACE

This is the fifth publication in a series of compilations of the reports on research completed for the Iowa State Highway Commission. This research was done for the Iowa State Highway Research Board Project HR-1, *"The Loess and Glacial Till Materials of Iowa; an Investigation of Their Physical and Chemical Properties and Techniques for Processing Them to Increase Their All-Weather Stability for Road Construction."* The research, started in 1950, was done by the Iowa Engineering Experiment Station under its project 283-S. The project was supported by funds from the Iowa State Highway Commission.

The principal objectives of the project may be summed up as follows:

1. To determine by means of both field and laboratory studies the areal and stratigraphic variation in the physical and chemical properties of the loess and glacial till materials of Iowa.
2. To develop new equipment and methods for evaluating physical and chemical properties of soil where needed.
3. To correlate fundamental soil properties with the performance of soils in the highway structure.
4. To develop a scientific approach to the problem of soil stabilization based on the relationships between the properties of the soils and those of the admixtures.
5. To determine the manner in which the loess and glacial till materials of Iowa can be processed for optimum performance as highway embankments, sub-grades, base courses, and surface courses.

Many of the papers in this bulletin were prepared originally as graduate theses required for master or doctoral degrees. Each was then re-written with the assistance of other project workers and was submitted to the Iowa Highway Research Board as a report on a phase of completed research. This explains the several authors for each paper. The research work was all done under Dr. D. T. Davidson as project leader in charge.

Practically all the papers herein have been published previously. The title page for each manuscript identifies all authors and gives the place and date of first publication. No attempt has been made to revise, update, and change the data; hence some contradictions are evident. The facts and conclusions presented are those of the authors at the time the manuscript was submitted. Much of the repetition of material has been eliminated, and the papers have been arranged by subject matter.

The list of REFERENCES at the end of each manuscript gives only the first or original printing, though the paper referred to may have appeared later in various forms in several publications, and some are included herein. Those shown as theses in the Iowa State University Library are so indicated because only in the theses are all the data shown.

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# EVALUATION OF LIME FOR STABILIZATION OF LOESS

by

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(American Society for Testing Materials Proceedings 56:1301-1315. 1960.)

## ABSTRACT

In lime stabilization of soils it is recognized that different soils vary quite widely, and treatments are adjusted accordingly. It has been reassuring to the soil engineer that he could look on lime as lime and not have to worry about it. Research now shows that some limes are more effective than others, and data in this paper emphasize the need for testing and evaluating lime to determine its optimum use. Test results with lime stabilized loess are related to Ca:Mg ratio in the lime and to the use of quicklime vs. hydrated lime. On the basis of compressive strength, quicklime appears to be more effective with loess than the equivalent amount of hydrated lime, and dolomitic lime is more effective than calcitic lime. The greater effectiveness of dolomitic lime is also indicated in artificial lime mixtures. Data are given comparing the CBR, plasticity indices, and shrinkage limits obtained after treatment with hydrated lime and quicklime. A high-temperature curing study is also included.

## INTRODUCTION

Loess of Wisconsin geologic age in Iowa and adjacent states is a major surficial material, and a method for treating loess to make it suitable for road base course construction would be highly desirable. With this objective in mind, research has recently been conducted in Iowa with lime. One of the first field tests of lime stabilization was a lime and loess road constructed in the 1920's[7], in southwest Iowa. Partly due to the lack of a wearing surface that road failed. The laboratory work described in this paper extends over the past several years and re-examines the feasibility of lime loess construction in southwest Iowa. Samples of loess were carefully chosen to represent the friable to plastic range, and tests were conducted using a number of varieties of lime. In the comparisons some interesting differences were found, particularly with reference to the lime, and contrasts could be made

between quicklime and hydrated lime, and between calcitic and dolomitic lime. The purpose of this paper is thus twofold: to resolve the question of the suitability of lime for loess stabilization and to point up the variable effectiveness of different limes. The mechanism of lime stabilization of loess is also discussed.

## MATERIALS

### Properties of Loess:

In Iowa the loess varies texturally from silty loam to silty clay. Clay mineral compositions are believed to be the same qualitatively[6], the principal clay mineral being an interlayer mixture of montmorillonite and illite. Cation exchange capacity is approximately proportional to the clay content.

The thick loess occurring near river valley and till plain source areas is characteristically calcareous and low in clay. Farther away from the sources the loess deposits are thinner, leached of carbonates, and higher in clay.

TABLE I. BRIEF DESCRIPTION OF LOESS SAMPLES

| Property | Sample No. 20-2 (IV) | 44A-1 |
|---|---|---|
| Location | Harrison Co. | Fremont Co. |
| Geologic material | Friable loess | Plastic loess leached |
| Thickness | Over 100 ft. | 15-20 ft. |
| Soil series | Hamburg | Marshall |
| Horizon | C | C |
| Engineering classification (AASHO) | A-4 (8) | A-7-6 (18) |

TABLE II. PROPERTIES OF LOESS SAMPLES

| Property | | Sample No. 20-2(IV) | 44A-1 |
|---|---|---|---|
| Textural composition*, percent by weight | Gravel | 0 | 0 |
| | Sand | 0.7 | 0.2 |
| | Silt | 78.3 | 58.0 |
| | Clay | 21.0 | 41.8 |
| | Colloidal | 15.8 | 31.0 |
| Textural classification† | | Silty clay loam | Silty clay |
| Physical properties | L.L., % | 32.9 | 53.1 |
| | P.L., % | 21.1 | 25.7 |
| | P.I., % | 11.7 | 27.4 |
| | S.L., % | 28.3 | 19.9 |
| | C.M.E., % | 15.2 | ND** |
| | F.M.E., % | 26.4 | ND |
| | Sp.Gr.‡ 25C/4C | 2.68 | ND |
| Predominent clay mineral | | Nontronite illite interlayer | Nontronite illite interlayer |
| Predominant exchangeable cations | | Calcium | Calcium |

| | | | |
|---|---|---|---|
| Chemical properties | Cation exchange cap. m.e./100 g | 13.4 | 28.2 |
| | pH‡ | 7.8 | 6.2 |
| | Carbonates§, % | 10.2 | ND |
| | Organic matter§, % | 0.2 | ND |

* Gravel—above 2.0 mm., sand—2.0 to 0.074 mm., silt—0.074 to 0.005 mm., clay—below 0.005 mm., colloidal—below 0.001 mm.

† Textural classification is based on the U. S. Bureau of Public Roads system except that 0.074 mm. was used as the lower limit of the sand fraction.

‡ Determined on the fraction passing No. 10 sieve.

§ Determined on the fraction passing No. 40 sieve.

**Not determined.

Two samples of C horizon loess were used in the lime stabilization studies; sample No. 20-2(IV) was selected to represent the friable, calcareous loess, and sample No. 44A-1 to represent the plastic, leached loess. Properties of the two samples, which for simplicity are referred to as the friable loess and the plastic loess, are given (tables I, II).

**Properties of Lime:**

Different commercial limes were furnished by the National Lime Association and include calcitic and dolomitic hydrated limes and quicklimes which meet the various standards of the association[7] (tables III, IV). Hydrated dolomitic limes are of the Type N (Monohydrate) in which magnesium occurs as the oxide and Type S (Dihydrate) in which magnesium is the hydroxide.

TABLE. III. PROPERTIES OF HYDRATED LIME USED

| Property | | Sample No. and Type of Lime | | | | | |
|---|---|---|---|---|---|---|---|
| | | H-1 calcitic | H-2 calcitic | H-3 dolomitic dihydrate | H-4 calcitic | H-5 calcitic | H-6 dolomitic monohydrate |
| Fineness, percent | Material passing No. 200 sieve | ND | 99.50 | ND | 99.4 | 99.0 | 99.55 |
| | Material passing No. 325 sieve | 99.0 | 98.75 | ND | ND | 98.4 | 99.24 |
| Chemical analysis, percent | Silicon dioxide | 0.80 | 0.25 | 0.57 | 0.52 | 0.58 | 0.6 |
| | Iron and aluminum oxide | 0.82 | 0.26 | 1.06 | 0.18 | 0.44 | 1.1 |
| | Magnesium oxide | 0.49 | 0.17 | 29.72 | 0.59 | 0.49 | 33.2 |
| | Calcium carbonate | 0.77 | ND | ND | 1.41 | 1.50 | ND |
| | Total calcium hydroxide | 97.82 | ND | ND | ND | 96.46 | ND |
| | Available calcium hydroxide | 97.38 | 94.85 | ND | 94.58 | ND | ND |
| | Loss on ignition | 24.56 | 23.15 | 27.06 | 24.52 | ND | ND |
| | Total calcium figured as CaO | ND | 74.53 | ND | ND | ND | ND |
| | Calcium oxide | ND | ND | 42.21 | 74.21 | 73.00 | 48.3 |
| | Combined $H_2O$ | ND | 22.83 | ND | ND | 0.45 | 16.8 |
| | Carbon dioxide | ND | ND | 2.90 | 0.62 | ND | ND |
| | Sulphur trioxide | ND | ND | 0.105 | ND | ND | ND |
| | Ca:Mg ratio | 181:1 | 512:1 | 1.7:1 | 146:1 | 180:1 | 1.7:1 |

TABLE IV. PROPERTIES OF QUICKLIME USED

| Property | | Sample No. and Type of Lime | | | | | |
|---|---|---|---|---|---|---|---|
| | | Q-1 calci-tic | Q-2 calci-tic | Q-3 dolo-mitic | Q-4 calci-tic | Q-5 calci-tic | Q-6 dolo-mitic |
| Fineness, percent | Material passing No. 50 sieve | 100.0 | 100.0 | ND | ND | ND | 99.98 |
| | Material passing No. 200 sieve | ND | 78.31 | ND | ND | ND | 97.01 |
| | Material passing No. 325 sieve | ND | 71.15 | ND | ND | 99.0 | ND |
| Chemical analysis, percent | Silicon dioxide | 1.97 | 0.34 | 0.67 | 0.38 | 0.75 | 0.7 |
| | Iron and aluminum oxide | 1.17 | 0.30 | 1.00 | 0.28 | 0.53 | 1.4 |
| | Magnesium oxide | 0.84 | 0.14 | 40.41 | 0.68 | 0.65 | 39.8 |
| | Calcium carbonate | 7.94 | ND | ND | ND | 2.76 | ND |
| | Total calcium oxide | 96.02 | ND | 56.59 | 96.45 | 95.00 | 58.1 |
| | Available calcium oxide | 92.52 | 93.80 | ND | 94.20 | ND | ND |
| | Loss on ignition | 4.45 | 0.66 | 1.70 | 2.03 | ND | ND |
| | Total calcium figured as CaO | ND | 97.75 | ND | ND | ND | ND |
| | Total sulphur | ND | ND | 0.037 | ND | ND | ND |
| | Carbon dioxide | ND | ND | ND | 1.27 | ND | ND |
| | Calcium sulfate | ND | ND | ND | ND | 0.10 | ND |
| | Ca:Mg ratio | 132:1 | 837:1 | 1.67:1 | 164:1 | 174:1 | 1.7:1 |

## OPTIMUM MOISTURE CONTENT

Prior to the main testing program, the optimum moisture content for maximum density was compared with the optimum moisture content for maximum strength by plotting the strength density and the moisture density relationships and reading the highest points. Specimens were molded with 6 percent hydrated lime H-1 and with the approximate equivalent of quicklime Q-1, 4.5 percent. The difference between optimum moisture contents for compaction and for strength was 2 or 3 percent with the hydrated lime, the higher moisture content giving lower density and highest strength. However, the difference in strength at the two moisture contents is slight: of the order of 4 to 8 percent. With quicklime the differences are even less, and for convenience the optimum moisture content for maximum density was used in the subsequent testing program.

## COMPRESSIVE STRENGTH

### Method

Each lime was added to the soil dry, the batch was mixed, and water was added. Specimens 2 in. in diameter and 2 in. high (h/d = 1) were then molded to a density of not less than 95 percent of standard Proctor density (ASTM Designation: 698-42T). A drop hammer molding apparatus was used; three specimens were molded for each combination of lime and loess. Specimens were cured 7 days in a cabinet maintained at 70° ± 3° F. and a relative humidity of not less than 90 percent. The specimens were then soaked in distilled water for 24 hours prior to testing; compressive strengths were determined using a load applied at 0.10 in. per minute.

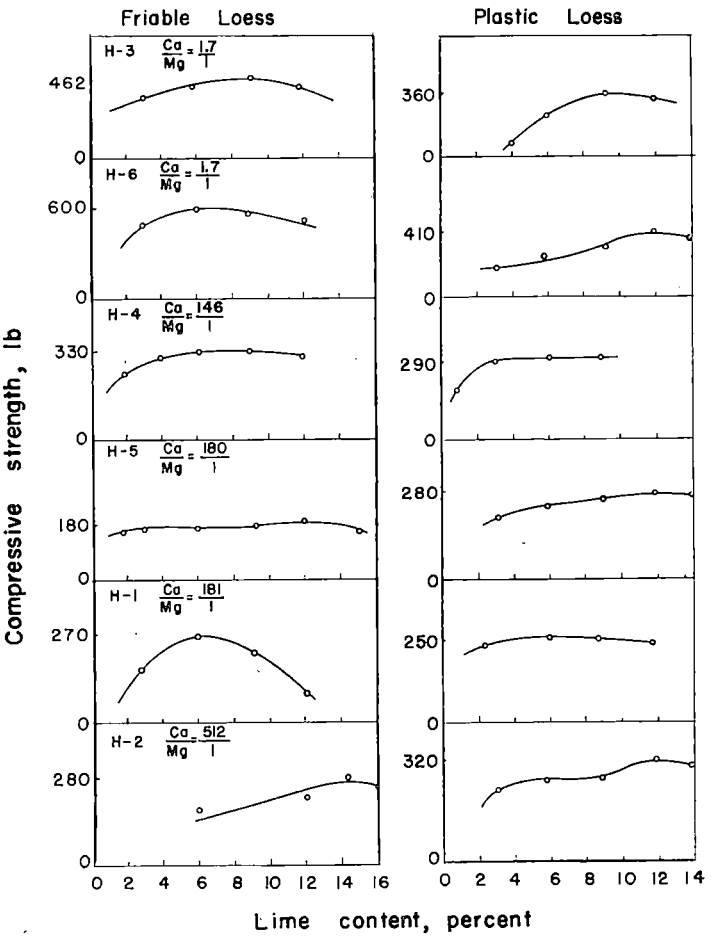


Fig. 1. Effect of hydrated lime content on the compressive strength of loess.

## Results

The change in compressive strength of lime stabilized loess with various percentages of different limes is shown graphically (figures 1, 2). All specimens with zero percent lime failed on immersion in water. The curves are considerably different in shape. Quicklimes show a decided optimum content which varies from 2.5 to 9 percent lime. The curve peaks tend to be less pronounced for the plastic loess. Some hydrated limes show compressive strength peaks; some do not. Curves which do not give decided peaks usually reach their strength quickly and then show a flattening out above a lime content of about 3 percent.

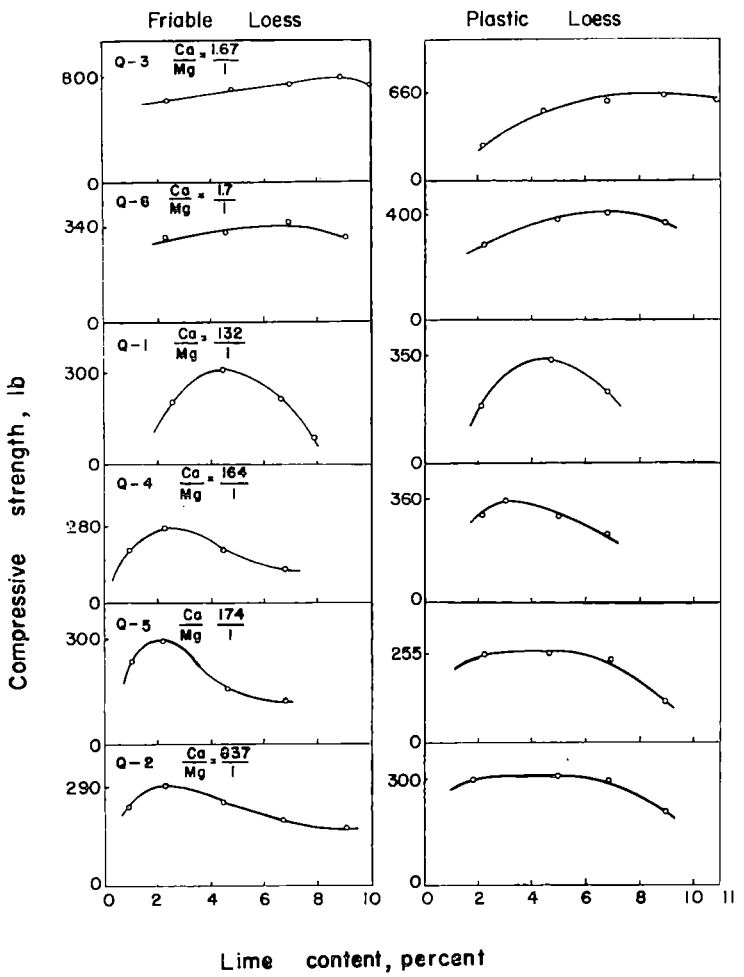


Fig. 2. Effect of quicklime content on the compressive strength of loess.

## Chemically equivalent percentages of various limes

To evaluate the different limes on a more or less equivalent basis, amounts were calculated which could give the same number of calcium plus magnesium ions. Allowances were made both for hydration and for difference in atomic weights. For example, 1.0 g of CaO (molecular weight 56.08) is equivalent to 1.32 g $Ca(OH)_2$ (molecular weight 74.11). In a dolomitic lime, 1.0 g of MgO (molecular weight 40.32) is equivalent to 1.84 g of $Ca(AH)_2$. Hydrated dolomitic lime is assumed to be made up of $Ca(OH)_2$ and MgO (table V). Strengths were interpolated from the lime content strength curves.

TABLE V. RELATION OF IMMERSED COMPRESSIVE STRENGTHS OF LIME STABILIZED LOESS TO PERCENT AND KIND OF LIME

| Properties of lime | | | Results with friable loess | | | | Results with plastic loess | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | 7-day compressive strength, lb. | | | | 7-day compressive strength, lb. | | | |
| Sample No. | Ca:Mg ion ratio | Wt. equiv. to 1 g $Ca(OH)_2$ | 3% (equiv.) lime* | 6% (equiv.) lime* | Opt. % lime | Opt. % lime | 3% (equiv.) lime* | 6% (equiv.) lime* | Opt. % lime | Opt. % lime |
| Hydrated Lime | | | | | | | | | | |
| H-3 | 1.7 | 0.905 | 240 | 265 | 462 | 9 | 35 | 275 | 358 | 9 |
| H-6 | 1.7 | 0.801 | 425 | 580 | 600 | 6 | 140 | 220 | 413 | 12 |
| H-4 | 146 | 1.01 | 200 | 207 | 335 | 6 | 208 | 210 | 290 | 6 |
| H-5 | 180 | 1.03 | 170 | 170 | 177 | 12 | 195 | 240 | 283 | 12 |
| H-1 | 181 | 1.01 | 210 | 270 | 270 | 6 | 240 | 250 | 250 | 6 |
| H-2 | 512 | 1.01 | 155 | 165 | 268 | 15 | 230 | 265 | 320 | 12 |
| Quicklime | | | | | | | | | | |
| Q-3 | 1.67 | 0.664 | 620 | 695 | 800 | 9 | 150 | 485 | 660 | 9 |
| Q-6 | 1.7 | 0.671 | 295 | 300 | 344 | 6.5 | 265 | 355 | 400 | 7 |
| Q-1 | 132 | 0.779 | 260 | 300 | 300 | 4.5 | 290 | 360 | 360 | 4.5 |
| Q-4 | 164 | 0.779 | 280 | 195 | 280 | 2.5 | 305 | 305 | 358 | 3 |
| Q-5 | 174 | 0.790 | 295 | 145 | 290 | 2.3 | 255 | 250 | 255 | 3 |
| Q-2 | 837 | 0.772 | 290 | 245 | 294 | 2.5 | 305 | 310 | 310 | 4.5 |

*Equivalent on an ionic basis to 3% or 6% $Ca(OH)_2$.

## Discussion

It is possible to add too much lime (figures 1, 2). Probably the excess lime acts as a filler separating the mineral grains, since hydrated lime crystals have a weak claylike (gibbsite) structure. Probably with continued curing the excess lime would be utilized, adding to strength. Characteristics of individual curves are probably related to individualities of each lime.

The data show that quicklime is in general more effective than hydrated lime (table V). With equivalent amounts the average strength is about 40 percent higher with quicklime. However, exceptions were found. The difference is not believed due to the heat of hydration of the quicklime, since the heat is rapidly dissipated during mixing and immediately after molding. Further work is under way to check this.

Another trend revealed is that dolomitic limes give higher strengths than calcitic limes. At equivalent percentages the effect is not so noticeable, but the strengths at higher percentages are quite different. Dolomitic limes can be used in higher percentages before there is a decrease in strength. Of the hydrated dolomitic limes, the monohydrate (N type) gives higher strength than the dihydrate (S type). However, the different brands of dolomitic quicklime also show variations in strength, and strengths with one dolomitic quicklime are double the strengths with the other. No explanation for this was found in hydration treatment or Ca:Mg ratio.

The above points out benefits to be derived from testing different limes before selecting one for a stabilized soil construction. With some combinations the same strength can be realized with one-half or one-third as much

lime. This sensitivity of soil to variables in lime should not be entirely unexpected; soils are chemically active and complex. A rational analysis may eventually become possible, but a major obstacle is the lack of adequate data on lime. Particle size and surface chemical factors related to curing are two qualities that need expression. When these qualities become measureable and measured, more definite correlations may be found and applied in the quality control of lime. Until then, laboratory tests on mixes of soil and lime seem to be the only answer.

With reference to differences in soils, the data show higher strengths with the friable loess (table V). This is not exactly as expected, since lime is supposed to react best with clays. The significance of this is discussed later under mechanism of lime stabilization.

### Artificial Lime Mixtures

To gain further evidence for some of the relationships discussed above, artificial dolomitic limes were prepared by mixing C. P. Reagent quality $Ca(OH)_2$ and MgO in various Ca:Mg ratios from 1:9 to 9:1 (figures 3, 4). With both the friable and the plastic loess the ideal lime composition appears to be between 1:1 and 3:1 Ca to Mg. Strengths dropped when MgO was increased beyond a ratio of 1:1.

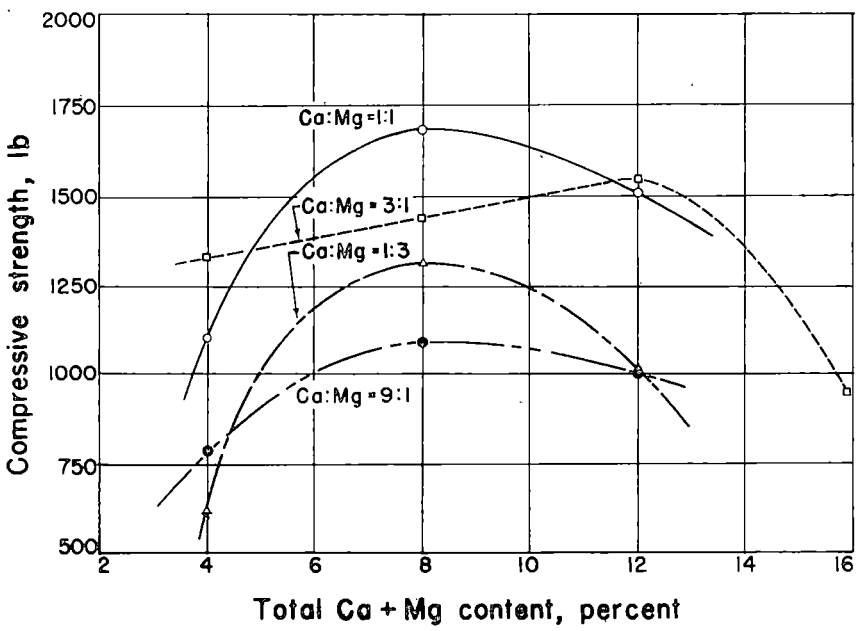


Fig. 3. Effect of Ca:Mg ratio on lime stabilization of friable loess.

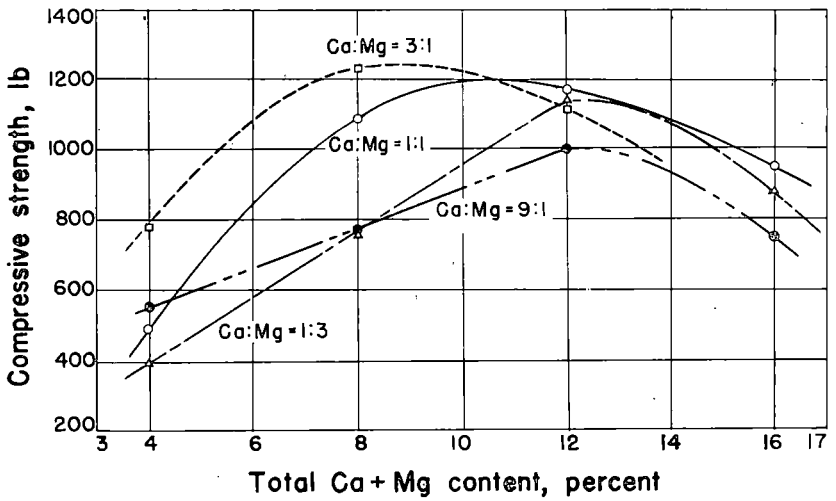


Fig. 4. Effect of Ca:Mg ratio on lime stabilization of plastic loess.

## CURING STUDIES

Among the variables which affect the strength of lime stabilized loess, curing is most important. Its effect on strength is a function of time, temperature, and relative humidity. Curing studies were conducted on loess samples stabilized with limes H-1 and Q-1. Specimens 2 by 2 inches were molded, cured, immersed in water for 24 hours, and tested for unconfined compressive strength. Various curing methods were used; in some methods specimens were wrapped in wax paper and aluminum foil and sealed with tape to prevent evaporation of moisture from the specimens and also prevent contact with carbon dioxide in the air. The oven cured specimens were also sealed in tin cans. The curing methods are as follows:

1. Wrapped and oven cured at 140° F.
2. Wrapped and oven cured at 110° F.
3. Wrapped and cured at 70° ± 3° F in a moist cabinet, relative humidity 90 percent.
4. Cured at 70° ± 3° F in a moist cabinet, relative humidity 90 percent. Specimens not wrapped.
5. Cured at 70° ± 3° F in a cabinet with relative humidity 45 percent. Specimens not wrapped.

### Effect of time

The strength of the lime stabilized loess increases rapidly at first, then increases more slowly at a more or less constant rate (figures 5, 6). After about fifteen weeks the increase is very slight or negative.

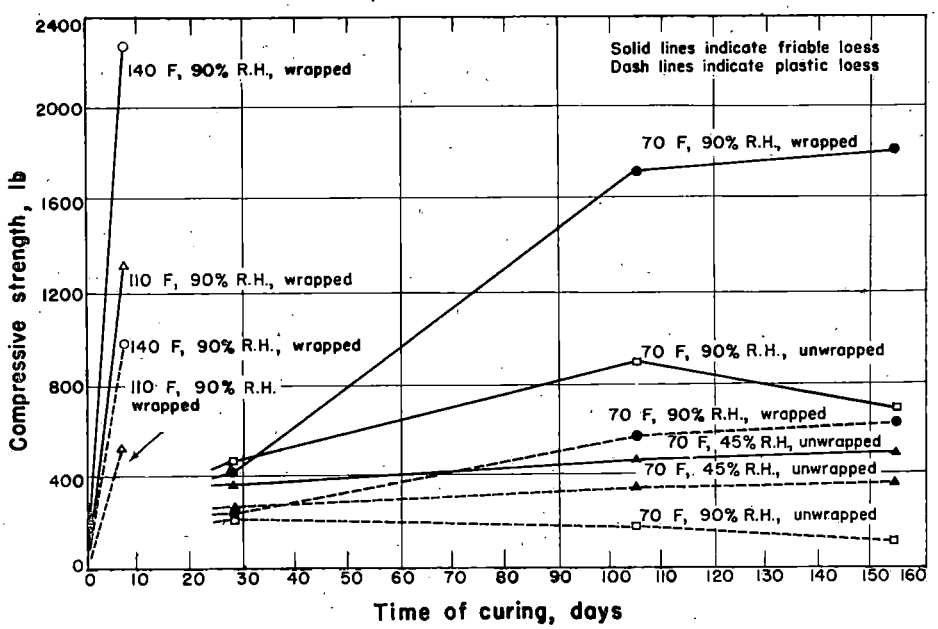


Fig. 5. Effect of curing on the compressive strength of loess stabilized with hydrated lime H-1.

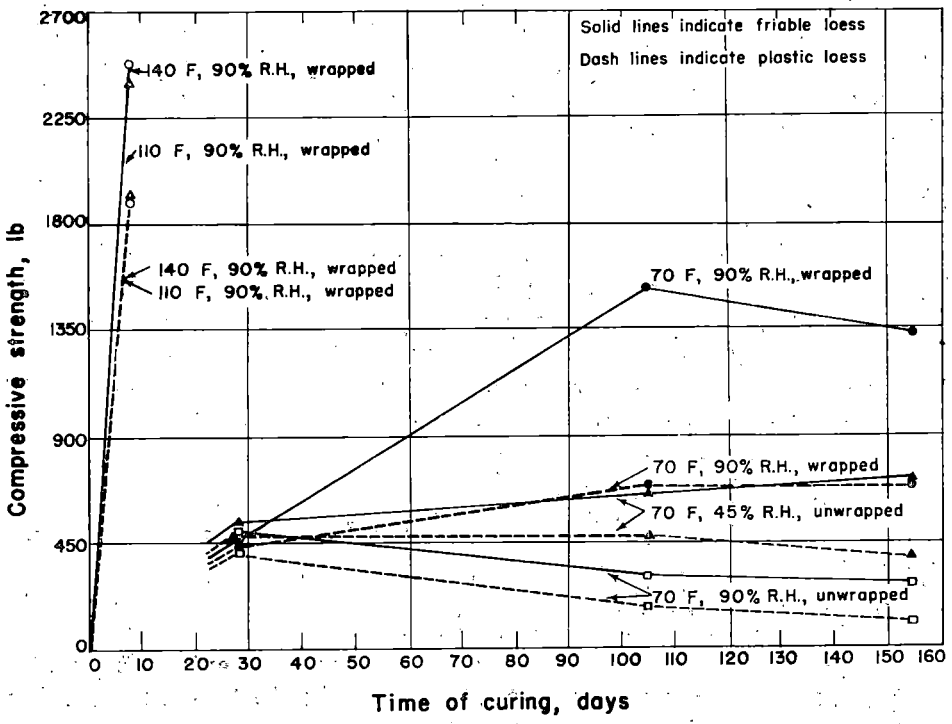


Fig. 6. Effect of curing on compressive strength of loess stabilized with quicklime Q-1.

**Effect of temperature**

The temperature of curing influences strength greatly. Seven day strengths after curing at 110° F or 140° F show a three to eight-fold increase in compressive strength over specimens cured at room temperature. With the plastic loess, 7 day strengths after curing at 110° F are reached in approximately 80 days curing at 70° F. With the friable loess the 110° F strengths are never reached by normal curing. No 140° F strengths could be duplicated by prolonged curing at room temperature. Therefore, accelerated curing, while perhaps useful to indicate relative reactivity, is not recommended for design purposes.

An interesting possibility is that decreased solubility of lime at higher temperatures may account for at least part of the strength increase. Heating a saturated lime solution from 70° F to 110° F will cause a precipitation of 17 percent of the dissolved lime, and heating to 140° F will cause precipitation of an additional 12 percent. However, the solubility at 70° F is only 0.164 g per 100 g of water, and heating an ordinary 200 g 2 by 2 inch lime soil specimen to 140° F will cause precipitation of only about 0.05 g of lime. It seems doubtful if the mechanical strengthening from this precipitation would cause an eightfold increase in strength, especially since lime crystals have a weak platy structure. Perhaps the precipitation would give better lime soil grain contacts and enhance chemical reactions. If this is true, the same effect should be found from alternate wetting and drying of stabilized specimens. The other possibility is that the heat effect is merely a matter of chemical reaction kinetics.

**Effect of Humidity**

All specimens wrapped and cured at 90 percent relative humidity show the greatest ultimate strength gain. It should be noted that this curing procedure would most nearly represent field curing conditions under an asphalt seal coat. The strength difference sometimes is not apparent until after 28 days, indicating that in damp weather there would be no hurry about applying the seal, and there would be little point in temporarily moist curing with wet burlap or straw.

Compressive strengths of unwrapped specimens were usually lower and less predictable. The extreme sensitivity to curing temperature may account for some of the irregularities. In some specimens 90 percent humidity gave higher strengths, in others 45 percent gave higher. Formation of carbonate on the surface of the specimen may aid in sealing the pores against moisture loss; calcium carbonate crusts formed in the surface layers of specimens after aging about a month. Crusts were observed in specimens molded with sand after curing only three days.

## CALIFORNIA BEARING RATIO

CBR tests were performed with standard AASHO compacted density and according to specifications of the U. S. Corps of Engineers[2]. Tests were run immediately after molding, after soaking 4 days, and after moist curing (90 percent humidity, 70° F) for 7 to 28 days (table VI). Data are yet

TABLE VI. CBR VALUES OF LIME STABILIZED LOESS

| | Results with friable loess | | | | Results with plastic loess | | | |
|---|---|---|---|---|---|---|---|---|
| Curing period | 0 | 0 | 7 | 28 days | 0 | 0 | 7 | 28 days |
| Condition at test: | As Molded | Soaked 4 days | | | As Molded | Soaked 4 days | | |
| Admixtures: | | | | | | | | |
| No admixture | 12.7 | 9 | 9 | .... | 10.5 | 5 | .... | .... |
| Calcitic lime: | | | | | | | | |
| 6% H-1 | 31.5 | 55 | .... | .... | 22.8 | 31 | .... | .... |
| 4.5% Q-1 | 34.0 | 39 | .... | .... | 24.0 | 39 | .... | .... |
| 3% Q-4 | .... | .... | 85 | 88 | .... | .... | .... | .... |
| Dolomitic lime: | | | | | | | | |
| 9% H-3 | .... | .... | 104 | 132 | .... | .... | .... | 66 |
| 9% Q-3 | .... | .... | 205 | 215 | .... | .... | 84 | 115 |

incomplete, so comparisons of different limes will not be made. The CBR values now available correlate well with 7 day compressive strength data, and it is believed the same trends will be realized. As an approximation, the CBR value of the friable loess after soaking is about one-fourth the unconfined compressive strength of the 2 by 2 in. cylinders; the CBR of the plastic loess after soaking is about one-eighth the compressive strength in pounds.

A considerable gain in CBR is indicated immediately after molding, probably due to cation exchange (table VI). The "as-molded" values are increased two to three times over those of the compacted raw loess. Soaking 4 days further increased the CBR of the lime treated specimens while decreasing the CBR of the raw loess. Soaking of the lime treated specimens for 4 days is believed to be an effective cure. An additional increase in CBR is evident after moist curing for 7 to 28 days. These strength gains perhaps illustrate the large benefits to be derived from the pozzolanic reaction. There was very little or no swelling in the CBR specimens during soaking.

The CBR values now available indicate that loess stabilized with lime would have suitable bearing capacity for use as a base course. Additional criteria are resistance to wetting and drying, and resistance to freeze-thaw. Two by two cylinders were found to gain strength through wetting and drying up to four cycles, probably since the added moisture benefits the cure. Submission of 28 day lime stabilized specimens to the standard wet-dry test for soil cement resulted in failure usually by volume change (swelling). Friable loess stabilized with 4.5 percent quicklime Q-1 showed no adverse volume change and withstood five cycles before the weight loss by brushing exceeded 10 percent. The resistance of lime stabilized loess to volume change is believed to be greatly influenced by confinement. Lime

stabilized loess, confined as it would be in a road, apparently has little tendency to swell, as indicated by measurements during the 4 day immersion of the CBR test, in which a 10 pound surcharge is used.

According to PCA criteria[8] adopted for soil cement, the freeze-thaw resistance of lime stabilized loess is very low. Specimens usually fail by weight loss during the first two or three cycles. Loss was not by flaking or spalling, as is often true with soil cement, but by brushing of material from the specimens. Since cementation of the lime stabilized specimens is relatively low, it is believed that brushing may be too severe a test. Work with lime fly ash stabilized soils lends support to this; lime, fly ash, and soil mixes which do not pass the brushing test have given satisfactory field performance.

## LIME MODIFICATION OF LOESS

The effects of equivalent amounts of limes H-1 and Q-1 on the pH, Atterberg limits, and standard Proctor density were measured immediately after mixing and after 7 days moist curing (table VII). The plasticity index

TABLE VII. MODIFICATION OF LOESS PROPERTIES BY ADDITION OF HYDRATED LIME OR THE EQUIVALENT AMOUNT OF QUICKLIME

| | Results with friable loess | | | | | Results with plastic loess | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Curing Time | 0 days | | | 7 days | | 0 days | | | 7 days | |
| Lime | 0 | 6% H-1 | 4.5% Q-1 | 6% H-1 | 4.5% Q-1 | 0 | 6% H-1 | 4.5% Q-1 | 6% H-1 | 4.5% Q-1 |
| LL, percent | 33 | 34 | 33 | 35 | 33 | 53 | 46 | 41 | 47 | 40 |
| PL, percent | 21 | 24 | 24 | 25 | 25 | 26 | 30 | 30 | 29 | 29 |
| PI, percent | 12 | 10 | 9 | 10 | 8 | 27 | 16 | 11 | 18 | 11 |
| SL, percent | 28 | 29 | 31 | 31 | 31 | 20 | 28 | 32 | 34 | 33 |
| pH | 7.8 | 12.6 | 12.6 | 12.1 | 12.0 | 6.2 | 12.5 | 12.7 | 11.8 | 12.0 |
| Max. dry density lb. per cu. ft. | 109.9 | 99.5 | 97.4 | .... | .... | 102.8 | 94.6 | 95.1 | .... | .... |
| Opt. moisture cont., percent | 18.2 | 17.1 | 18.1 | .... | .... | 21.4 | 20.7 | 21.7 | .... | .... |

of the friable loess is only slightly reduced by the addition of lime; the plasticity index of the plastic loess shows a comparatively large reduction. The quicklime was more effective at reducing the plasticity index of both loess samples, although with the friable loess sample the difference is actually of little practical significance. The shrinkage limit was increased by the addition of lime; the plastic soil showed the greatest increase. Quicklime was somewhat more effective. On mixing lime with the loess, pH values took an immediate jump to about 12.6 but were reduced during curing, suggesting lime depletion through reactions between loess and lime.

Hydrated lime by increasing aggregation decreases the maximum compacted density. The flocculation effect decreases the optimum moisture content about one percent. Quicklime causes about the same reduction in maximum density but raises the optimum moisture content back to its previous level, since water is immediately used in the lime hydration. In the mixes given, 1.5 percent moisture would be sufficient to convert the quicklime.

## REACTION OF LIME WITH LOESS

It is generally recognized that lime can improve the properties of certain soils through cation exchange. However, the natural cations in the loess samples investigated are calcium, and the flocculation mechanism does not appear to be so much an ion exchange as a crowding of additional ions on the clay. This would lower the zeta potential and produce a flocculation. (Since the zeta potential is the effective negative charge at the surface of the clay, the addition of positive ions will reduce the charge.) An immediate flocculation effect is observed during mixing and is reflected in lowering of the liquid limit, raising of the plastic and shrinkage limits, and lowering the maximum compacted density.

In addition to the immediate improvement in properties brought about by flocculation, there is a long-term strength gain in lime stabilized specimens. The reason for this has been long speculated on[1, 3, 7], and it appears certain that there is an additional reaction besides flocculation or cation exchange. The high sensitivity of strength gain to the curing temperature suggests a chemical reaction. Carbonation of the lime is important[3]; however, specimens wrapped and sealed against the entry of $CO_2$ gave strengths as high or higher than specimens left unwrapped. Unwrapped specimens definitely showed the development of a carbonate rich layer around the outside.

For long-time strength gain a pozzolanic reaction has been suggested, and it is usually supposed that this reaction takes place with the clay. However, in this investigation higher strengths were realized with less clay, and there was little difference in optimum lime content between high clay and low clay. The same trend has been reported; a montmorillonite containing 40 to 50 percent nonclay minerals gave stabilometer values several times those obtained with a nearly pure montmorillonitic clay. These percentages of clay are so high as to minimize the influences of granulometry; a soil containing 50 percent clay would allow little contact between the silt grains. This would suggest that important reactions may occur between lime and the non-clay minerals. Petrographic and soil cement studies with loess show that strength correlates with quartz surface area[5], and data soon to be published verify this. It, therefore, appears that pozzolanic reactions may be most pronounced with the non-clay minerals and particularly with quartz, so long as the particle size is fine enough[4].

## CONCLUSIONS

The laboratory data indicate that a field trial of lime stabilized western Iowa loess would be desirable. However, the laboratory results point out the desirability of evaluating a lime prior to its use for stabilization. With Iowa loess a dolomitic lime would probably be more effective. Quicklime, though requiring more caution in handling, may offer the best and most economical way for lime stabilization. It is not yet known if tank truck hydration of the quicklime will yield the same results as mixing it dry with the loess. With proper information on the lime and on the soil, the most desirable properties of lime for a certain job may eventually be predicted; it appears that the main deterrent at the present time is lack of information on the lime. A simplified pozzolanic reaction rate test would therefore seem desirable.

Resistance of lime stabilized loess to freezing and thawing can best be evaluated by means of field trials, after which suitable laboratory test criteria might be developed. Curing is best at high temperatures, so early summer construction seems desirable. Moist conditions should be maintained for proper curing, for example, by the use of a bituminous seal applied immediately after finishing the base. Strengths attained in the laboratory by accelerated or high temperature curing may never be reached under normal field conditions and so should not be used in design.

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# THE CALCIUM:MAGNESIUM RATIO IN SOIL LIME STABILIZATION

by

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## ABSTRACT

Preliminary studies of soil lime stabilization with loess pointed up the fact that different limes give widely varying strength results, and strengths seemed to be related to Ca:Mg ratio and to the use of quicklime vs. hydrated lime. Now additional data are available verifying this fact for loess, glacial till, dune sand, and an alluvial backswamp clay. Mixtures of pure chemicals were used to certify the trends, and results generally supported those obtained with commercial limes. Dolomitic lime usually gives higher strengths, particularly when added in amounts greater than 4 percent by weight of the soil. The dihydrate dolomitic lime (Type S) prepared by autoclave process is not so effective as the monohydrate (Type N) in which Mg occurs as the oxide. This also was reaffirmed using pure chemicals. Highest strengths were realized with dolomitic quicklime, the strength advantage over other limes being 50 to 100 percent or more with 6 or 8 percent admixture. The difference was found to depend on the kind of soil, but the difference was significant with all soils tested.

Practical value of the soil lime results is emphasized by the CBR and wet-dry and freeze-thaw data. CBR values for lime stabilized loess and till were frequently doubled by use of dolomitic quicklime, and CBR's with 4 to 9 percent dolomitic lime ran from 100 to over 200 after 28 days moist curing. In these and other tests all molded specimens were wrapped to prevent killing of the lime by $CO_2$ from the atmosphere.

Use of dolomitic lime gives the first important indication of soil lime having a satisfactory resistance to weathering. Stabilized loess and till specimens cured 1 to 12 weeks were found in many cases to gain strength through 12 cycles of wet-dry. Freeze-thaw specimens showed only slight decrease in strength or in some cases gained strength through 12 cycles. The best results were again obtained with dolomitic quicklime. Freeze-thaw results with calcitic lime were much less favorable, and most specimens lost strength rapidly after the first few cycles.

## INTRODUCTION

Recent research done on soil lime stabilization[3] showed that limes differ in effectiveness in their reaction with loess, a common silt material in the central U.S. This difference in cementation appeared to be related mainly to chemical composition of the lime. Objectives of the present research were to gain more information to further illuminate this problem, and to prove or disprove the superiority of certain limes not only for loess, but for a sand, a glacial till, and an alluvial clay as well.

### The Ca:Mg ratio

Commercial lime is produced by crushing and calcining limestone to drive off $CO_2$, leaving the calcium oxide or quicklime. Many producers also burn dolomite, a carbonate rock similar to limestone but containing magnesium. The resulting lime is calcium oxide plus magnesium oxide, and is termed dolomitic quicklime. Differences in the two types of limes were recently recognized in the plaster industry and have been given emphasis in the past few years. The term *calcitic* is taken from calcite, the common $CaCO_3$ mineral in limestones. The mineral dolomite is $CaMg(CO_3)_2$. Unless dolomitic quicklime is given special hydration treatment, the high calcium or calcitic lime is preferred to prevent cracking. Calcium oxide hydrates quickly, but magnesium oxide at normal temperatures hydrates very slowly, and hydration over the years produces expansion and cracking of walls[2]. At present this is eliminated by a forced autoclave hydration of the magnesium oxide, converting it to hydroxide.

There are two chemical types of quicklime: Calcitic, $CaO$; and dolomitic, $CaO + MgO$. Three chemical classes of hydrated lime are: Calcitic, $Ca(OH)_2$; dolomitic Type N (normal) also called monohydrate, $Ca(OH)_2 + MgO$; and dolomitic Type S (steam), also called dihydrate, $Ca(OH)_2 + Mg(OH)_2$. The degree to which a lime is calcitic or dolomitic can be expressed in the calcium to magnesium mole ratio, Ca/Mg. In pure calcite, $CaCO_3$, the ratio is infinity; in pure dolomite, $CaMg(CO_3)_2$, it is one.

## MATERIALS

### Soil materials

A brief description of the soil samples is given in table I. Properties of these soils are given in table II.

*Loess.* Of the five soil samples studied two are loess samples from western Iowa. Loess is a silty soil material showing systematic areal variations in particle size and soil profile development. The two samples represent a friable, calcareous C horizon loess classifying as a silty clay loam, and a plastic, noncalcareous C horizon loess classifying as silty clay. The major clay minerals are montmorillonite and illite.

*Glacial till.* A C horizon sample from southern Iowa was selected as repre-

TABLE I. THE SOILS USED

| Soil: | Friable loess (Lab. No. 20-2) | Plastic loess (Lab. No. 44A-1) | Glacial (Lab. No. 411) | Alluvial clay (Lab. No. Al-1) | Dune sand (Lab. No. S-6-2) |
|---|---|---|---|---|---|
| Location: | Harrison County, SW Iowa | Page County, SW Iowa | Page County, SW Iowa | Woodbury County, W Iowa | Benton County, E Iowa |
| Geological description: | Wisconsin age friable loess, oxidized. Thickness over 100 ft. | Wisconsin plastic loess, oxidized, leached. Total thickness 15-20 ft. | Kansan glacial till, oxidized calcareous. | Recent Backswamp clay from Missouri River. | Wisconsin eolian sand fine-grained, oxidized, leached. |
| Soil series: | Hamburg | Marshall | Shelby | Luton | Carrington |
| Horizon: | C | C | C | C | C |
| Sampling depth, ft.: | 39 - 40 | 4 - 5 | 3 - 23 | | 6 - 11 |

TABLE II. PROPERTIES OF SOILS USED

| Soil: | Friable loess | Plastic loess | Glacial till | Alluvial clay | Dune sand |
|---|---|---|---|---|---|
| Textural composition, %* | | | | | |
| Gravel (2.0 mm.) | 0 | 0 | 0 | 0 | 0 |
| Sand (2.0 — 0.074 mm.) | 0.4 | 0.2 | 25.2 | 1.5 | 94.4 |
| Silt (74 — 5$\mu$) | 82.2 | 58.0 | 31.8 | 24.2 | 1.6 |
| Clay (< 5$\mu$) | 17.4 | 41.8 | 43.0 | 74.3 | 4.0 |
| Colloids (< 1$\mu$) | 12.5 | 31.0 | 32.5 | 55.1 | 3.5 |
| Predominant clay mineral† | Montmorillonite and illite | Montmorillonite and illite | Montmorillonite and illite | Montmorillonite and illite | Montmorillonite and illite |
| Probable predominant exchangeable cation: | Calcium | Calcium | Calcium | Calcium | .... |
| Specific gravity 25°c/4°C | 2.68 | 2.72 | 2.66 | | 2.64 |
| Chemical properties: | | | | | |
| Cat. ex. cap., m.e./100 gm‡ | 13.4 | 28.2 | 20.0 | 39.4 | |
| Carbonates,§ % | 10.5 | 0 | | | 0 |
| pH | 7.8 | 6.2 | 7.9 | 7.3 | 6.5 |
| Organic matter | 0.2 | 0.5 | 0.1 | 1.7 | 0.04 |
| Physical properties: | | | | | |
| Liquid limit, % | 32.9 | 53.1 | 41.8 | 71.0 | 19.0 |
| Plastic limit, % | 21.1 | 25.7 | 14.9 | 24.5 | .... |
| Plasticity index | 11.8 | 27.4 | 26.9 | 46.5 | N.P. |
| Shrinkage limit, % | 22.0 | 19.9 | 12.3 | 10.2 | .... |
| Centrifuge moist. equiv. % | 19.0 | 21.3 | 23.9 | 48.7 | .... |
| Field moist. equiv., % | 26.4 | 53.6 | 31.0 | 38.4 | .... |
| Classification: | | | | | |
| Textural | Silty clay loam | Silty clay | Clay | Clay | Sand |
| Engineering (A.A.S.H.O.) | A-4(8) | A-7-6(18) | A-7-6(15) | A-7-6(20) | A-3(0) |

*Dispersed by air-jet with sodium metaphosphate dispersing agent
†From differential thermal analysis of fraction passing No. 200 sieve
‡Fraction passing No. 40 sieve
§From differential thermal analysis

sentative of Kansan age calcareous glacial till. The till sample classifies texturally as a clay but contains significant amounts of sand and silt. The predominant clay minerals are montmorillonite and illite.

*Alluvial clay.* A sample of alluvial clay from the Missouri River floodplain in western Iowa represents recent flood or overflow sediments denoted

geologically as backswamp deposits or slack water sediments. Weathering is in its initial stages, and the clay is calcareous. A rather high organic matter content suggests that recently eroded topsoil has been incorporated in the deposit. From differential thermal analysis the major clay minerals appear to be montmorillonite and illite.

*Dune sand.* The sand sample is from one of the local deposits of stable dune or eolian sands on the Iowa (early Wisconsin) drift plain in eastern Iowa. The sample is C horizon, being taken from below the major soil profile. The sand is rather fine and nearly all one size, therefore poor for compaction. A small percentage of clay occurs as grain coatings.

### Lime

Commercial limes were selected to represent the various chemical classes. From the chemical analyses furnished by the lime producers the Ca:Mg ratio is 1.7:1 in the dolomitic limes; in the calcitic limes it is 147:1 and 168:1 (table III).

TABLE III. CHEMICAL ANALYSES OF COMMERCIAL LIMES USED

| | Calcitic lime | | Dolomitic lime | | |
|---|---|---|---|---|---|
| Kind of lime: | Quicklime | Hydrated | Quick-lime | Mono-hydrate | Dihy-drate |
| Silicon dioxide | 0.38 | 0.52 | 0.67 | 0.6 | 0.57 |
| Iron and aluminum oxide | 0.28 | 0.18 | 1.00 | 1.1 | 1.06 |
| Total calcium figured as CaO | 96.45 | 74.21 | 56.59 | 48.3 | 42.21 |
| Total magnesium figured as MgO | 0.68 | 0.59 | 40.41 | 33.2 | 29.72 |
| Total sulphur | ND* | ND | 0.04 | ND | ND |
| Sulphur trioxide | ND | ND | ND | ND | 0.11 |
| Carbon dioxide | 1.27 | 0.62 | ND | ND | 2.90 |
| Loss on ignition | 2.03 | 24.52 | 1.70 | ND | 27.06 |
| Ca:Mg ratio | 168:1 | 147:1 | 1.7:1 | 1.7:1 | 1.7:1 |

*Not determined

Synthetic limes were prepared from purified grade $Ca(OH)_2$, $Mg(OH)_2$ and MgO. These were mixed in various proportions to duplicate the three classes of commercial hydrated lime.

## METHODS

### Mixing and molding

Soil lime mixes were proportioned and mixed dry; then optimum water content for maximum density was added, and the materials were machine mixed for 5 minutes. Most specimens were then molded immediately, but some of the sand quicklime mixes were allowed to hydrate for various times before molding. A double plunger drophammer molding apparatus was used to mold 2 by 2 inch specimens to approximate standard Proctor den-

sity. C.B.R. specimens also were prepared at standard Proctor density[5].

**Curing**

Curing was at 70°F ± 3°F and with a relative humidity of not less than 90 percent. The curing time was varied. Specimens for certain investigations such as long curing were double wrapped in waxed paper and aluminum foil to preserve moisture better and to prevent entry of carbon dioxide from the air.

**Compressive strength testing**

At the end of the specified curing period some specimens were tested immediately, but most were immersed in distilled water for one day. Specimens were tested for unconfined compressive strength using a load travel of 0.10 inch per minute. Tests were run in triplicate and the average strengths were recorded.

**C.B.R. testing**

C.B.R. tests were run after specimens were soaked in water for four days. The C.B.R. readings were made at 0.1″ and 0.2″ penetration and the larger value reported.

**Tests for durability.**

Cured 2 by 2 inch specimens were subjected to alternate wetting and drying or to alternate freezing and thawing. Each period of immersion wetting and air drying was for one day. Freezing was for 23 hours at −10° F; thawing was in the open air for two hours and in the moist cure cabinet for 23 hours. In either sequence a cycle takes two days. Specimens were tested for compressive strength after 1, 3, 6, 9, and 12 cycles wetting and drying or freezing and thawing.

## SYNTHETIC LIMES

Use of synthetic limes made from prepared chemicals allows isolation of the Ca:Mg ratio as an independent variable. Other work has shown close correlations between results with chemicals and those with commercial limes[3]. In the present work the Ca:Mg ratio was varied between the two end limits, and mixtures were prepared to represent both the monohydrate and dihydrate types of hydrated dolomitic lime. CaO quicklime was not included in the study.

Typical results with synthetic hydrated lime mixtures and one soil in this research and in others show that highest 28 day strengths were obtained with about 6 to 8 percent lime (figures 1, 2). Apparently excess lime

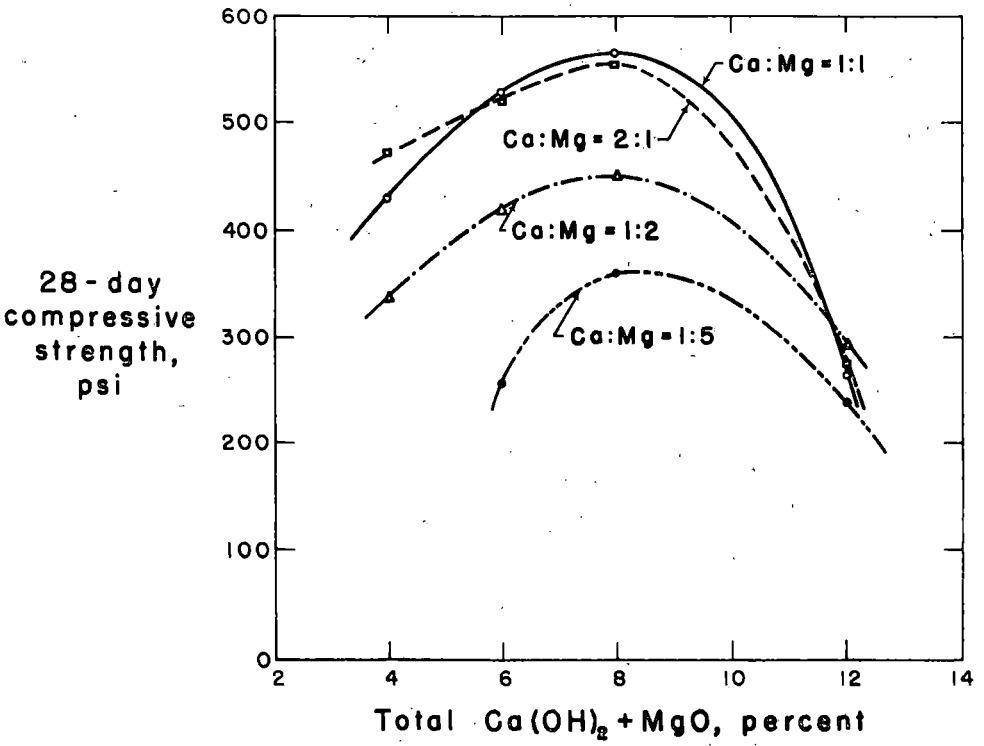


Fig. 1. Immersed strengths of glacial till stabilized with limes synthesized from $Ca(OH)_2$ and MgO.

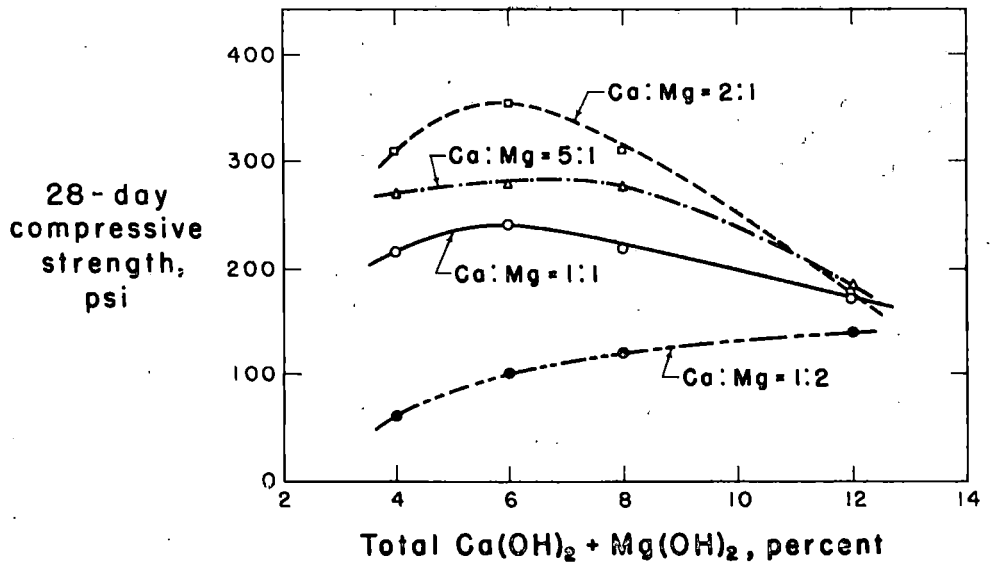


Fig. 2. Immersed strengths of gracial till stabilized with limes synthesized from $Ca(OH)_2$ and $Mg(OH)_2$.

can act as a weak filler, subtracting from strength.

The relation between strength and Ca:Mg ratio may be illustrated by plotting strengths at one lime content. Strengths with 8 percent lime and varying Ca:Mg ratio are shown (figures 3, 4, 5).

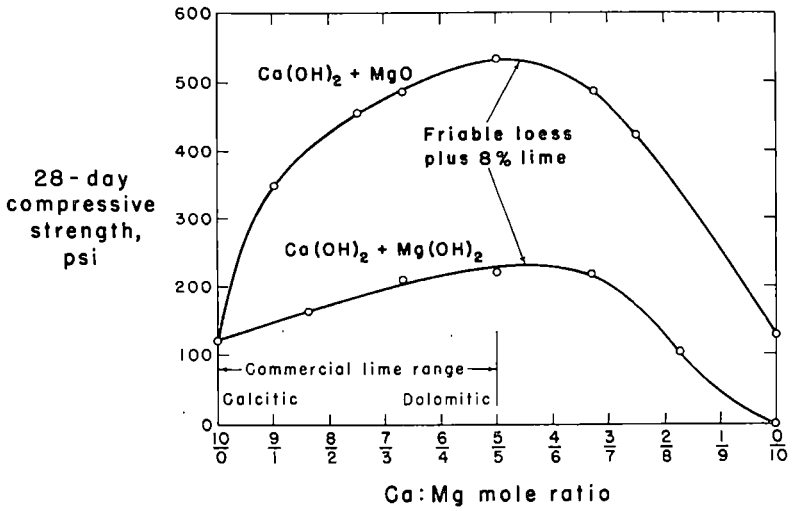


**Fig. 3. Effect of Ca:Mg ratio on immersed strength of friable loess with 8 percent synthetic hydrated lime.**

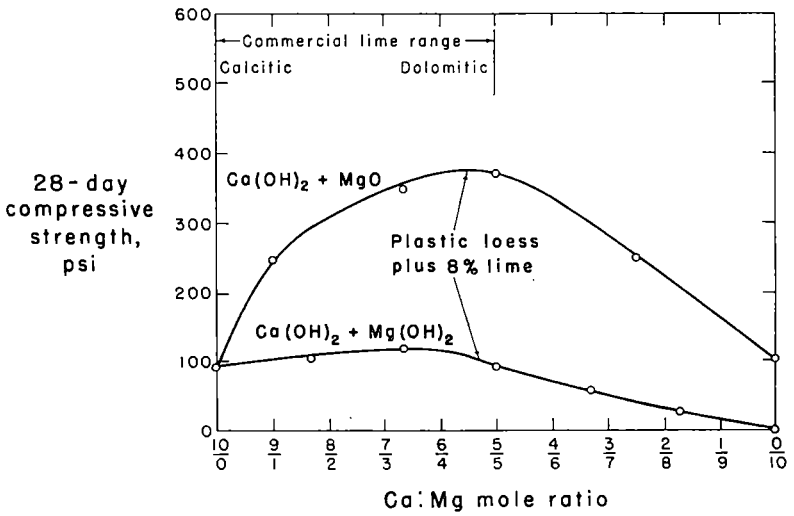


**Fig. 4. Effect of Ca:Mg ratio on immersed strength of plastic loess with 8 percent synthetic hydrated lime.**

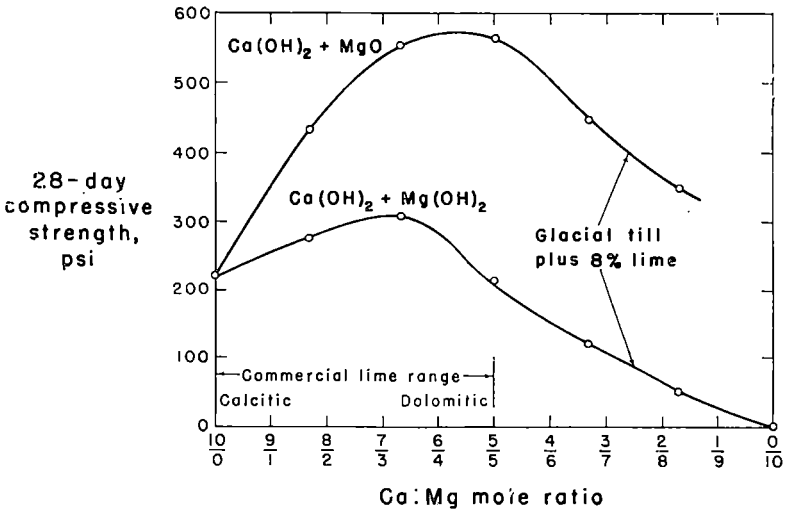


**Fig. 5. Effect of Ca:Mg ratio on immersed strength of glacial till with 8 percent synthetic hydrated lime.**

Strengths tend to be higher with dolomitic lime, but the most significant increase is with the monohydrate. With monohydrate lime the optimum Ca:Mg ratio approximates that of pure dolomitic lime. A further increase of MgO is not possible with commercial limes, nor is it desirable, as strengths go down. It is of academic interest to note that some strength is realized with pure MgO, but specimens molded with pure $Mg(OH)_2$ slaked when put in water. Twenty eight day strengths with dolomitic monohydrate lime are from two to four times greater than those obtained with calcitic hydrated lime, the greatest difference being with lower clay content. The lime stabilized loesses give double or triple strength with only a very small addition of MgO. The glacial till has the highest clay content and is least sensitive to replacement of $Ca(OH)_2$ with MgO.

## COMMERCIAL LIMES

The seven day strength curves with different percentages of commercial limes reflect trends previously found with synthetic limes (figures 6, 7, 8). Monohydrate dolomitic lime gave higher strengths that dihydrate, and dihydrate gave higher strengths than calcitic lime except in the low lime content range, However, strength differences between mono and dihydrate are not nearly so pronounced as they were with synthetic chemical limes. Related to this is that strengths with monohydrate lime are not as high as they should be. For comparison, mixes were prepared using quicklime. Use of dolomitic quicklime raised the strengths up to those previously

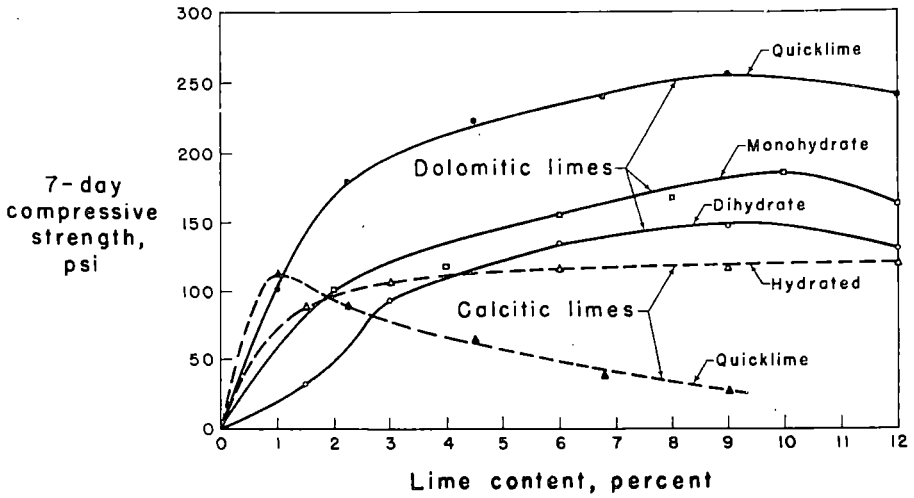


Fig. 6. Immersed strength of friable loess with commercial lime.

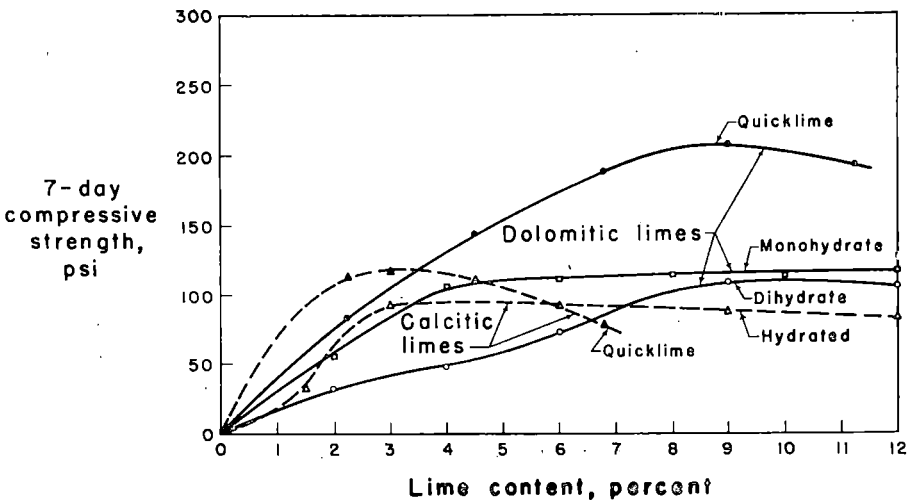


Fig. 7. Immersed strength of plastic loess with commercial lime.

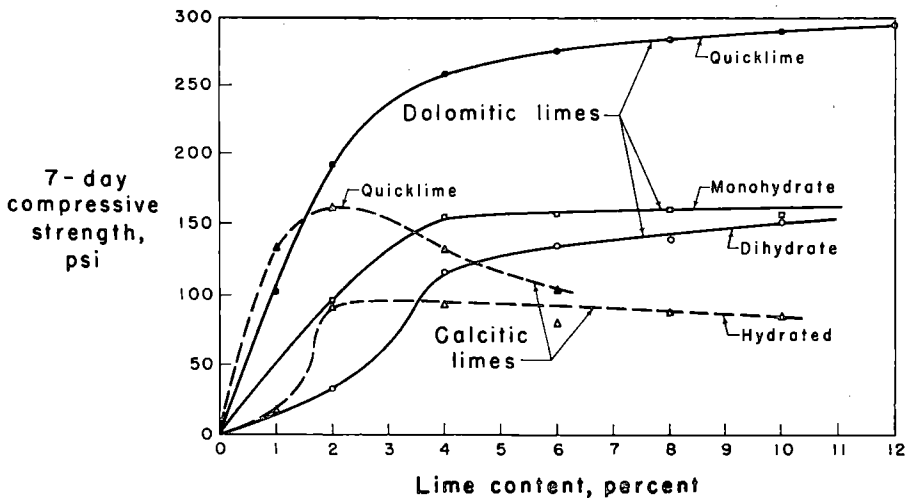


**Fig. 8. Immersed strength of glacial till with commercial lime.**

realized with the synthetic monohydrate lime (table IV). This is also true if lime contents are expressed on a molar equivalent basis.

TABLE IV. COMPARATIVE 28-DAY STRENGTHS WITH DIFFERENT KINDS OF DOLOMITIC LIME. LIME CONTENTS 8 TO 9 PERCENT.

| Lime | Immersed strength, psi | |
|---|---|---|
| | Friable loess | Plastic loess |
| Synthetic monohydrate | 506 | 356 |
| Commercial monohydrate | 274 | 122 |
| Quicklime | 588 | 312 |

That MgO is most effective when added as a pure chemical or in quicklime and its effectiveness is reduced in commercial monohydrate lime suggests a possible relation to the hydration process. Hydration of dolomitic quicklime after it is mixed with soil is a rather cold process, and in small batches the heat was quickly dissipated in the mixing bowl. There is probably little conversion of MgO. Commercial hydration involves much heat, depending on the process, and perhaps commercial processes result in MgO being coated with a thin layer of hydroxide, in effect partially killing the MgO in the lime. It is known that if heat and pressure are raised high enough MgO will completely convert to the hydroxide, as in the autoclave manufacture of dihydrate lime. This might explain various differences in effectiveness of different brands of monohydrate dolomitic lime.

Calcitic quicklime strengths are highest with about 1 to 3 percent lime, and quicklime strengths exceed those with calcitic hydrated lime. At higher lime contents the quicklime strengths drop off, the greatest drop being with the least clay. It is believed that hydration in the molded soil lime specimen may be a significant factor with these higher contents of calcitic lime. The factor is not so much one of heat as of pressure built up by expansion of hydrating lime. In some specimens expansion and cracking were observed. The more friable samples, having less clay and lower cohesive tensile strength, would be most sensitive to this type of destruction.

The high strengths with calcitic quicklime in the low lime content range are rather difficult to explain. One possibility is that hydration and the accompanying forced crystal change in lime while it is being mixed with soil may benefit the soil lime interface contact and allow a more efficient use of lime for chemical bonding. Chemical precipitation of lime in lime soil mixtures as a result of higher temperature has been found to increase strengths of both lime[3] and soil, lime and fly ash, probably because of improved intimacy of grain contact[1].

Use of dolomitic and calcitic quicklime with an alluvial clay and a dune sand was investigated (figure 9). Highest strengths were with dolomitic

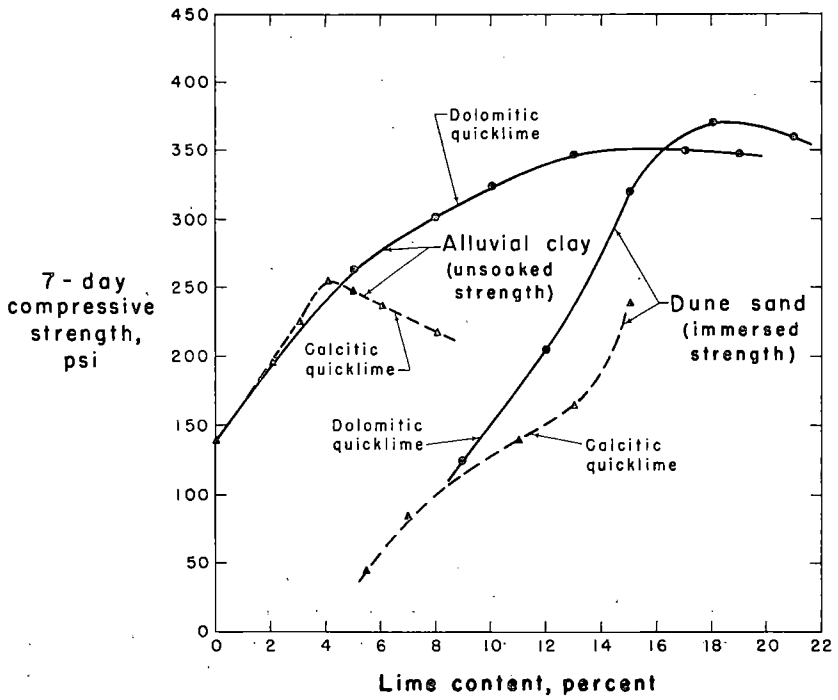


Fig. 9. Strengths of dune sand and of alluvial clay with commercial lime.

lime, although low percentages of calcitic quicklime gave high strengths with the clay. Results with the alluvial clay are not substantially different from those with the other clayey soils. Results with the sand show a necessity for high lime contents, probably because of the lack of fine grained material to give suitable grain contact area for cementation.

## DURABILITY OF SOIL LIME

A durability test without brushing was used with soil lime: 2 by 2 inch specimens were subjected to 12 cycles of wet-dry or freeze-thaw, and the final compressive strength was measured. This strength was compared to that obtained from specimens moist cured to the same total age but not subjected to wet-dry or freeze-thaw. The reference specimens were immersed in water one day prior to testing to duplicate the saturated condition of the weathered specimens. The result of weathering can be expressed as a durability ratio, or the ratio of strength after weathering to the strength of reference specimens the same age[4]. A durability ratio of 1.0 indicates no adverse effect from weathering; a durability ratio of 0.0 denotes failure due to weathering.

TABLE V. WET-DRY AND FREEZE-THAW DURABILITY RATIOS OF SOIL LIME

Durability ratio = $\frac{\text{Strength after 12 weathering cycles}}{\text{Immersed strength after moist curing to same age}}$

| Soil Material | Kind of lime | % lime | Moist cure, days 7 + 12 cycles wet-dry | 28 | 56 | Moist cure, days 7 + 12 cycles freeze-thaw* | 28 | 56 |
|---|---|---|---|---|---|---|---|---|
| Friable loess | Calc. hyd. | 3 | 0.3 | 0.4 | 1.0 | 0 | 0 | 0 |
| | Calc. quick. | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| | Dol. dihyd. | 9 | 1.0 | 0.7 | 1.0 | 0 | 0 | 0 |
| | Dol. quick. | 9 | 0.6 | 0.9 | 1.0 | 0.4 | 0.7 | 0.8 |
| Plastic loess | Calc. hyd. | 3 | 0 | 0.7 | 1.0 | 0 | 0 | 0 |
| | Calc. quick. | 3 | 0.2 | 0.8 | 0.7 | 0 | 0 | 0 |
| | Dol. dihyd. | 9 | 0 | 0.3 | 0.3 | 0 | 0 | 0 |
| | Dol. quick. | 9 | 0.7 | 0.8 | 0.9 | 0.1 | 0.4 | 0.6 |
| Glacial till | Calc. Hyd. | 2 | 0 | 0 | 0 | 0 | 0 | 0 |
| | Calc. quick. | 2 | 0 | 0 | 0 | 0 | 0 | 0.2 |
| | Dol. dihyd. | 4 | 0 | 0.1 | 0.2 | 0 | 0.1 | 0.2 |
| | Dol. quick. | 4 | 0.5 | 0.4 | 0.4 | 0.5 | 0.4 | 0.4 |

*Total age is moist cure time plus 24 days.

Results from durability tests are shown in table V. Lime contents for these tests were selected on the basis of 7 day compressive strength curves (figures 6, 7, 8) and represent percentages which either gave maximum or near maximum 7 day strength. The only lime which gives an indication of satisfactory weathering resistance to both wet-dry and freeze-thaw is dolomitic quicklime, which also gave highest compressive strength. Dihy-

drate dolomitic lime added in the same percentages usually gave lower durability. From compressive strength curves it would be anticipated that the monohydrate would give a durability in between, and calcitic limes added in the same amounts would give low durability. Lesser amounts of calcitic lime did give low durability but in some instances there was satisfactory resistance to wet-dry (table V). Many soil lime specimens gained strength through the cycles of wet-dry. Durability in general is increased by longer curing before the initiation of wet-dry or freeze-thaw cycles. Since freezing takes place with specimens resting on wet felt, the test is probably rather severe.

There also seems to be a relation between durability and kind of soil; the friable loess is most durable and the glacial till is least durable.

## C.B.R. OF SOIL LIME

The California Bearing Ratio was determined for the same soil and lime mixtures studied for durability (table VI). Highest C.B.R.'s were

TABLE VI. C.B.R. OF SOIL LIME AFTER VARIOUS PERIODS OF CURING AND 4-DAY SOAKING

| Soil material | Kind of lime | % lime | Pre-soak curing time, days 0 | 7 | 28 |
|---|---|---|---|---|---|
| Friable loess | .... | 0 | 9 | .... | .... |
| | Calc. hyd. | 3 | 24 | 57 | 77 |
| | Calc. quick. | 1 | 26 | 85 | 88 |
| | Dol. dihyd. | 9 | 23 | 104 | 132 |
| | Dol. quick. | 9 | 29 | 205 | 215 |
| Plastic loess | .... | 0 | 5 | .... | .... |
| | Calc. hyd. | 3 | 23 | 38 | 49 |
| | Calc. quick. | 3 | 38 | 46 | 61 |
| | Dol. dihyd. | 9 | 39 | 52 | 66 |
| | Dol. quick. | 9 | 49 | 84 | 115 |
| Glacial till | .... | 0 | 3 | .... | .... |
| | Calc. hyd. | 2 | 20 | 62 | 64 |
| | Calc. quick. | 2 | 46 | 105 | 115 |
| | Dol. dihyd. | 4 | 25 | 92 | 107 |
| | Dol. quick. | 4 | 48 | 120 | 178 |

gained with dolomitic quicklime. The C.B.R.'s increased with increased curing time, as would be expected from compressive strength data. For a given soil the C.B.R. is approximately proportional to the unconfined compressive strength. The same relationship is reported for soil cement[4]. There is little correlation between C.B.R. and durability ratio. Data indicate that a satisfactory C.B.R. can be realized after 11 days or less, and the numerical gain in C.B.R. from 11 to 32 days is only from 2 to 30.

## CONCLUSION

In conclusion it can be stated that lime cementation of soil at ordinary temperatures is best when the lime is a dolomitic monohydrate, of molar equal parts of $Ca(OH)_2$ and MgO. Except at very low lime contents any large departure from this composition resulted in lower strength and durability. Occurence of magnesium as the hydroxide, $Mg(OH)_2$, greatly reduces its effectiveness, and from strength data and comparisons with quicklimes it seems that the commercial monohydrate used may have endured partial hydration of its MgO.

With commercial limes the highest strengths were realized when dolomitic quicklime was added and allowed to hydrate with the soil, and strengths were approximately those previously reached with monohydrate lime made up from pure chemicals. It therefore appears that the commercial hydration process may have a very significant bearing on the effectiveness of dolomitic lime; dissipation of heat during hydration might prevent partial conversion of MgO to $Mg(OH)_2$.

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# POWDER VS. SLURRY APPLICATION OF LIME FOR SOIL STABILIZATION

by

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(American Society for Testing Materials Proceedings. 1959.)

## ABSTRACT

Both hydrated and quicklime have proved successful in soil stabilization. Quicklime is less expensive and more effective, but hydrated lime is easier and safer to handle in the powdered state. A possible answer to the safer use of quicklime as a soil stabilizer lies in its application in the form of a slurry.

The effectiveness of limes in slurry form when applied to silty and to clayey soils is evaluated, and the results are compared with stabilization by the use of limes in powdered form.

Slurries of lime were found to be easily applied and produced unconfined compressive strengths equivalent to strengths obtained through the use of powdered lime. Quicklimes in slurries produced higher strengths than those used in powdered form. Hydrated limes, powdered or in slurries, gave about equivalent strengths. Dolomitic limes were superior to calcitic limes. The amounts of either type of lime required for near maximum 7 and 28 day strengths were found to be economical.

## INTRODUCTION

Construction of stabilized roads with powdered lime may be hampered by health problems created by lime dust in the air, especially during hot windy weather. Lime is a caustic material that can cause damage to eyes and skin ranging from minor irritations to serious burns. Both hydrated lime and quicklime release heat upon contact with water, but quicklime releases much more heat because of its highly exothermic hydration reaction. The combination of heat and causticity causes destruction of living tissue. Other obvious objections to dusting are the loss of lime and the whitewashing of adjacent property.

Hand spreading of bagged lime or mechanical spreading of bulk lime have been adopted to minimize dusting during construction. Neither method

has been entirely satisfactory during windy weather. Lime slurries have been successfully spread through spray bars in Texas[2], but no data are available on the comparative effectiveness of lime applied as a slurry or as a powder. The laboratory investigation reported in this paper was undertaken to determine the effect of the form of lime application on the strengths of soil lime mixtures.

## MATERIALS USED

### Properties of soils

Two fine grained soils representative of major surficial deposits in Iowa, were selected for this investigation[4] (table I).

TABLE I. DESCRIPTION AND PROPERTIES OF SOIL SAMPLES

| | Friable loess (lab. no. 20-2V) | Kansan till (lab. no. 409-12C) |
|---|---|---|
| Geological description: | Wisconsin age loess, friable, calcareous | Kansan age glacial till, plastic, calcareous |
| Location: | Harrison County, Western Iowa | Ringgold County, S. W. Iowa |
| Soil Series: | Hamburg | Shelby |
| Horizon: | C | C |
| Sampling depth, ft.: | 39 - 40 | 4½ - 10½ |
| Textural composition, %: | | |
| Gravel (> 2 mm) | 0 | 3 |
| Sand (2 — 0.074 mm) | Trace | 31 |
| Silt (74 = 5 $\mu$) | 83 | 28 |
| Clay (< 5 $\mu$) | 17 | 38 |
| Colloids (< 1 $\mu$) | 12 | 30 |
| Predominant clay minerals* | Montmorillonite and illite | Montmorillonite and illite |
| Chemical properties: | | |
| Cat. ex. cap., m.e./gm† | 13.4 | 29.5 |
| Carbonates, % | 10.2 | 2.1 |
| pH‡ | 7.8 | 8.3 |
| Organic matter, % | 0.2 | 0.1 |
| Physical properties: | | |
| Liquid limit, % | 32.9 | 42.4 |
| Plastic limit, % | 21.1 | 20.5 |
| Plasticity index | 11.8 | 21.9 |
| Shrinkage limit, % | 28.3 | 8.2 |
| Classification: | | |
| Textural§ | Silty clay loam | Clay |
| Engineering (AASHO) | A-4(8) | A-7-6(12) |

*By X-ray diffraction analysis.

†For fraction passing No. 40 sieve.

‡For fraction passing No. 10 sieve.

§From triangular chart developed by U. S. Bureau of Public Roads, but 0.074 mm. was used as the lower limit of the sand fraction.

### Properties of limes

Four limes, representative of commercial calcitic (high calcium) and dolomitic quicklime and hydrated lime, were used (table II).

TABLE II. PROPERTIES OF LIMES USED

| | Quicklimes | | Hydrated limes | |
|---|---|---|---|---|
| | Dolomitic | Calcitic | Dolomitic | calcitic |
| Trade name: | Kemidol oxide | Kemikal oxide | Kemidol hydrated (monohydrate) | Kemikal hydrated |
| Processing location: | Genoa, Ohio | New Braunfels, Texas | Genoa, Ohio | New Braunfels, Texas |
| Chemical analysis, % by wt.: | | | | |
| Magnesium oxide | 37.9 | 0.5 | 31.8 | 0.6 |
| Calcium oxide | 59.3 | 95.6 | 48.8 | 73.8 |
| Silica | 0.5 | 1.1 | .... | 0.3 |
| Iron and aluminum oxide | 0.3 | 0.9 | .... | 0.6 |
| Sulphur trioxide | 1.1 | Trace | .... | 0.4 |
| Loss on ignition | 0.6 | 1.9 | .... | 24.1 |

## SAMPLE PREPARATION AND TESTING

### Consistency of lime slurries

Lime slurries are suspensions of lime in water, varying in consistency from a thin mobile suspension to a thick paste. In the field lime slurries are best applied by pressure controlled spraying through spray bars. This method of distribution limits the maximum usable concentration of a slurry.

Workable slurry concentrations were estimated by observations of a number of lime slurries of different concentrations. It appears that the maximum allowable concentrations for spraying are: 50 percent dolomitic hydrated lime, 50 percent calcitic hydrated lime, 40 percent dolomitic quicklime, and 30 percent calcitic quicklime, expressed as a percentage of the weight of water.

These maximum slurry concentrations limit the amount of lime that can be applied to soil in the field in a slurry form provided the optimum moisture content of the resulting soil lime mixture is not to be exceeded. Ten percent lime (based on dry soil) is the maximum lime percentage that can result from applying a 50 percent slurry to the soils used in this study, and a 6 percent maximum results from a 30 percent slurry.

### Density—strength—moisture relations

Mixtures of soil, lime, and water were prepared and tested to find the moisture content for maximum density and the mosture content for maximum 7 day strength. The data showed that the optimum moisture for maximum density and the optimum moisture for maximum immersed strength

closely correspond. The data also showed that optimum moisture varies mainly with lime content and slightly with type of lime. The form of application (slurry or powder) had little effect on the optimum moisture requirements.

Most specimens for testing except those with quicklime slurries were molded at optimum moisture for standard density[5]. Mixtures of soil and quicklime slurries were molded at varying moisture contents, and the strength corresponding to the optimum moisture content was interpolated from a graph of strength versus moisture content. The interpolation was necessary because of difficulty experienced in moisture content control due to non-uniform evaporation of water during slaking.

**Preparation of soil and powdered lime mixtures**

The air-dried soil was crushed to pass a No. 10 sieve. The desired amount of hydrated lime or quicklime was dry mixed with the soil for 20 seconds in a Hobart Model C-100 mixer at moderate speed. Distilled water was added to bring the mixture to the optimum moisture content, and the moistened mixture was machine mixed for 5 minutes. Soil, lime mixtures with lime contents of 0, 2, 4, 6, 8, and 10 percent of the dry weight of the soil were prepared.

Hydrated calcitic lime contents are expressed as percent $Ca(OH)_2$, hydrated dolomitic as percent $Ca(OH)_2$ and MgO, calcitic quicklime as percent CaO and dolomitic quicklime as percent CaO and MgO.

**Preparation of soil and slurry lime mixtures**

Lime slurries were prepared by adding the desired amount of hydrated lime to the amount of water needed to bring the soil lime mixture to optimum moisture content; with the quicklimes an additional amount of water was required to slake the quicklime. The slurry was mixed and allowed to slake for half an hour before it was mixed with the soil. No benefit was found from the use of longer soaking periods. The slurry and soil were mixed for 5 minutes. Mixtures having the same lime contents as those mixed with powdered lime were prepared.

**Molding of Specimens**

Immediately after mixing, 2 by 2 inch specimens were molded to near standard ASTM density by placing a predetermined amount of the mixture in a molding cylinder and compacting the mixture with five blows on each face by a 5 pound hammer dropped from a height of 12 inches. After removal from the molding cylinder, specimens were weighed to the nearest 0.1 gram and their heights were measured to the nearest 0.001 inch.

**Curing**

The specimens were sealed in waxed paper and allowed to cure in a

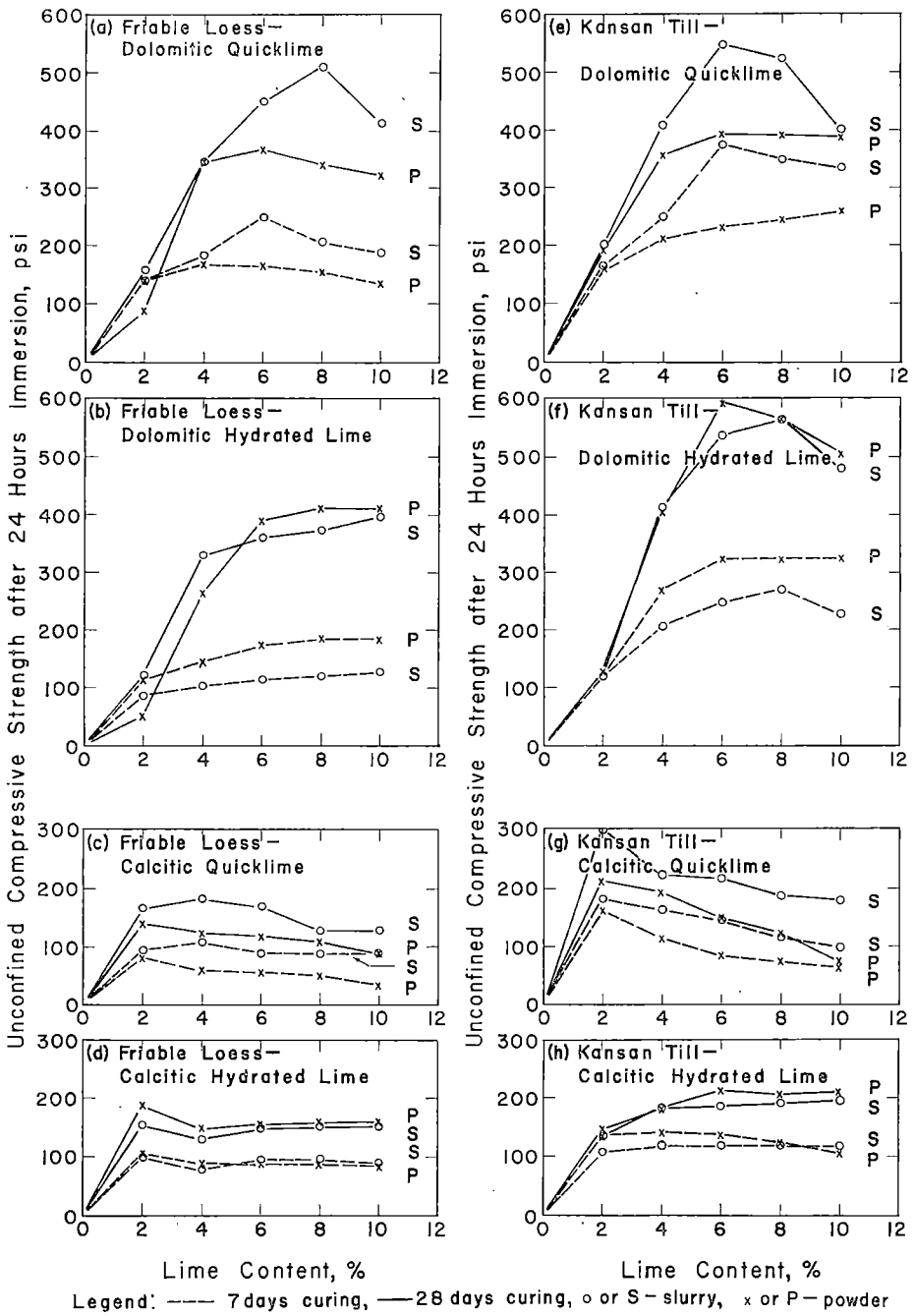


**Fig. 1. Effects of type, amount and form of application of lime on the unconfined compressive strength of lime stabilized soil tested after 24 hours immersion in distilled water.**

humidity room at temperature of 75±5° F and a relative humidity of not less than 90 percent. Three specimens from each mixture were cured for 7 days and three for 28 days.

### Compressive Strength Testing

Specimens were unwrapped after curing and soaked in distilled water for 24 hours. The height of each specimen was measured before and after immersion. Specimens were tested after immersion for unconfined compressive strength using a load travel of 0.10 inch per minute. Averages of three values of strengths are reported.

## TEST RESULTS

Unconfined compressive strength test results, both for soil and lime slurry mixtures and for soil and lime powder mixtures, are plotted of strength versus lime content (figure 1). Graphs *a, b, c, d* are for friable loess and graphs *e, f, g, h* are for Kansan till. The type of lime used is shown for each.

Comparison of equal curing time slurry and powder curves obtained with both soils shows that quicklime slurries gave higher strengths than quicklime powders. The hydrated lime strengths obtained with slurries and powders show that the powdered form results were slightly better in most cases.

Dolomitic limes gave markedly better strengths than calcitic limes, particularly after 28 days curing. With calcitic limes maximum strengths were obtained with about 2 percent lime. The use of 4 percent of either of the dolomitic limes gave strengths nearly double those obtainable with the calcitic limes. Maximum strengths with the dolomitic limes were obtained with 6 to 10 percent lime.

The quicklime slurries and hydrated limes in either form were about equally effective with a few exceptions (figure 1).

Strengths over 500 psi after 28 days curing were obtained with dolomitic lime in the following mixtures: friable loess and 8 percent quicklime applied as a slurry, Kansan till and 6 or 8 percent quicklime applied as a slurry, Kansan till and 6 or 8 percent hydrated lime applied as either a slurry or powder. Iowa Engineering Experiment Station experience has indicated that for soil lime stabilization strengths of about 500 psi are indicative of satisfactory freeze-thaw resistance.

The good results with the Kansan till are especially encouraging, since this soil represents one of the major problem soil types in the Midwest.

### Density

Standard ASTM density to lime content relationships show that the type of lime had little effect on the maximum density of soil lime mixtures,

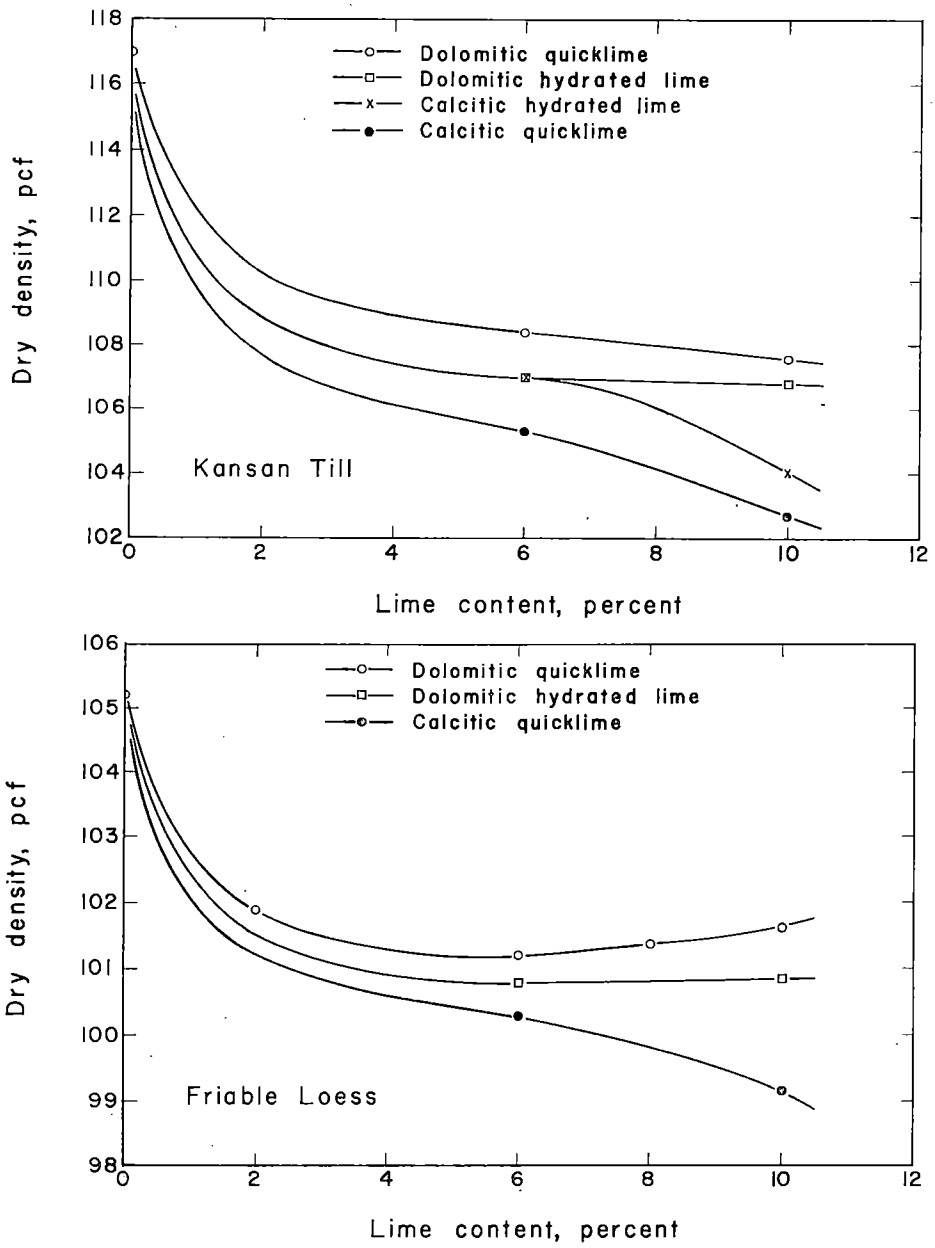


**Fig. 2. Effect of variation of lime content on maximum dry density.**

except at about 10 percent lime (figure 2). The method of lime application, slurry or powder, had even less effect on density (table III). A small addi-

TABLE III. SUMMARY OF MOISTURE-DENSITY AND MOISTURE-STRENGTH RELATIONSHIPS OF FRIABLE LOESS AND KANSAN TILL STABILIZED WITH DIFFERENT KINDS OF LIME.

| Type of lime | Lime content % | Form of lime application | Friable loess | | Kansan till | |
|---|---|---|---|---|---|---|
| | | | Optimum moisture content, % | Maximum dry density, pcf | Optimum moisture content, % | Maximum dry density, pcf |
| Dolomitic quicklime | 0 | — | 19.9 | 105.1 | 14.0 | 117.1 |
| | 2 | Slurry | 21.1 | 101.9 | .... | .... |
| | 2 | Powder | 21.1 | 101.9 | .... | .... |
| | 6 | Slurry | 21.2 | 101.2 | 17.1 | 108.4 |
| | 6 | Powder | 21.2 | 101.2 | .... | .... |
| | 8 | Slurry | 20.2 | 101.4 | .... | .... |
| | 10 | Slurry | 20.3 | 101.6 | 16.7 | 107.5 |
| | 10 | Powder | 20.3 | 101.6 | .... | .... |
| Dolomitic hydrated lime | 0 | .... | 19.9 | 105.1 | 14.9 | 117.1 |
| | 6 | Slurry | 20.8 | 100.8 | .... | .... |
| | 6 | Powder | .... | .... | 19.4 | 106.9 |
| | 10 | Slurry | 20.3 | 100.8 | .... | .... |
| | 10 | Powder | 20.3 | 100.8 | 19.3 | 106.3 |
| Calcitic quicklime | 0 | .... | 19.9 | 105.1 | 14.9 | 117.1 |
| | 6 | Slurry | 21.5 | 100.3 | 17.6 | 105.3 |
| | 6 | Powder | .... | .... | 17.6 | 105.3 |
| | 10 | Slurry | 22.2 | 99.2 | 18.0 | 102.8 |
| | 10 | Powder | 22.2 | 99.2 | .... | .... |
| Calcitic hydrated lime | 0 | .... | 19.9 | 105.1 | 14.9 | 117.1 |
| | 6 | Powder | .... | .... | 18.4 | 106.9 |
| | 10 | Powder | .... | .... | 20.0 | 104.1 |

tion of lime causes a marked decrease in density below that of the raw soil. Further additions of lime also cause a slight decrease in density.

## DISCUSSION

The speed of a chemical reaction depends on the nature and condition of the reactants and the surrounding medium in which the reaction occurs. Generally the more factors that are held constant the easier a study of chemical kinetics becomes. The following discussion of chemical phenomena involved in soil lime reactions, assuming constant temperature and moisture conditions, leads to an hypothesis for reactions in soil and lime mixtures.

The chemical changes resulting from contact of lime and soil minerals in the presence of water may be divided into two categories, ion exchange and pozzolanic reaction. Ion exchange includes all ions that take part in crowding action around clay particles as well as those that are exchanged for replaceable ions on the clay particles. With any one soil, the initial rate of the pozzolanic reaction at constant temperature ideally depends on the concentration of ionized lime in solution. The ionic concentration at any time depends on the rate at which depleted ions are replaced (rate of solubility). Ion exchange is relatively rapid and is probably completed before the pozzo-

lanic reaction starts. In fact, ion exchange is so rapid compared with the pozzolanic reaction that initially all calcium ions in solution are probably needed to complete the ion exchange. Therefore the pozzolanic reaction cannot start until the ion exchange is completed. But after that the only ionic factor limiting the rate of the pozzolanic reaction is the rate of solution. Little or no pozzolanic reaction products are formed if all the lime is consumed in ion exchange.

Calcium and hydroxyl ions begin to go into solution from the surfaces of hydrated lime crystals as soon as the crystals come in contact with water. Ions are also returned to the crystal structure from the solution at all times. The rate of solution is more rapid than the rate of crystallization until the solution becomes saturated with ions, then an equilibrium condition exists in which the two rates are equal. Equilibrium requires that the lime be present in both the dissolved and undissolved conditions.

The addition of water to quicklime is thought to cause a tightly cemented crust to form around aggregations of quicklime; the outside hull of the agglomerate becomes hydrated lime while the core remains quicklime. No aggregations are formed if quicklime is added to water. The formation of a crust retards the rate of slaking, and therefore equilibrium conditions may not be rapidly reached. The crust formed will no doubt be dissolved and the core slaked in due time. However, in the presence of soil and with the passage of time, pozzolanic reaction products form and ions resulting from quicklime cores may have become sealed off. At best, any further pozzolanic reaction will then depend on the rate of ion diffusion through the surrounding pozzolanic cement.

The hydration reaction of calcium oxide is highly exothermic and proceeds rapidly, but the hydration of magnisium oxide is slow and is promoted by heat. The heat liberated from the slaking reaction is rapidly dissipated through water when the reaction takes place in more water than is required for hydration. When the amount of water is near that required for hydration, the heat developed must be dissipated through the immediately surrounding material and may be locally concentrated. The crust formation is probably partially due to absorption of some of the heat of reaction. The excess heat may also be localized enough to cause some of the MgO in dolomitic lime to hydrate to $Mg(OH)_2$.

Previous studies have shown that MgO either as a component of the lime as in dolomitic lime or as an additive to calcitic lime increases the strength of lime stabilized soil[1, 3]. $Mg(OH)_2$ was not found to be beneficial for either. The exact mechanism of strength improvement with MgO is not yet understood. The better results are possibly due to MgO catalysis of the reaction between $Ca(OH)_2$ and pozzolanic minerals. However, the evidence shows that there is a definite molar ratio of Ca:Mg at which the strength due to cementation is a maximum. This suggests that MgO actually enters the pozzolanic reaction as a unit and becomes an integral part of

some cementation products which are more easily crystallized and have a more stable crystal structure than can be formed from $Ca(OH)_2$ without MgO. The reasoning implies that the pozzolanic reaction involving dolomitic lime would be more rapid than the reaction involving calcitic lime.

Flocculation effects due to the introduction of calcium ions have been observed and related to both cation exchange and crowding action of calcium ions around clay particles[1]. The flocculation effects due to calcium ions result in both a lowered plasticity index and a raised shrinkage limit, as well as a reduction of the maximum compacted density. Minimum lime additions to develop maximum flocculation effects have been found to be less than 3 percent, the exact percentage depending on the nature and amount of the clay minerals present.

The foregoing discussion of the physico chemical phenomena involved in soil lime stabilization aids in interpreting and explaining the results of this investigation.

The strengths obtained with the quicklime slurries were higher than those obtained with the quicklime powders, the poorer results with the powders probably being due to the formation of crusts around quicklime agglomerates in the soil mixture and to the partial hydration of the MgO (in the dolomitic quicklime) to form $Mg(OH)_2$. The hydrated limes were about equally effective when used in either the slurry or powder form, since no crust or $Mg(OH)_2$ formation occurs in either method of application. Probably for the same reasons, with a few exceptions the quicklime and hydrated lime slurries gave near equal results.

Strength and density are usually closely related, with increased density giving increased strength, and decreased density giving decreased strength. The data (figures 1, 2) show that immersed strengths increased with lime content and that the values of maximum density decreased, perhaps indicating that strength loss due to decreased density is more than compensated for by pozzolanic cementation. The decrease in density with increased lime contents up to about 2 percent is probably due to an increase in flocculation of clay particles. The slight density decrease with increased lime contents above 2 percent is most likely due to the difference in the specific gravity of soil and lime.

## CONCLUSIONS

The following conclusions concerning the form of lime application to soil are made on the basis of the best relative strengths resulting from various soil and lime mixtures:

(1) Quicklime should be applied only as a slurry.

(2) Hydrated lime may be applied either as a powder or as a slurry.

(3) Quicklime applied as a slurry and hydrated lime applied either as a powder or as a slurry are about equally effective for soil stabilization.

(4) The dolomitic limes give higher strengths than the calcitic limes, and the soils used can be satisfactorily stabilized with the dolomitic limes.

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## DISCUSSION

Chairman W. H. Goetz.*—I do not have a clear picture of the efficacy of hydrated lime and quicklime with respect to application in slurry form. Does it make any difference whether this hydration is completed in the slurry before it is added to the soil, or will hydration continue?

Mr. J. B. Sheeler (author).—The two materials give about equivalent results—the hydrated lime used either in a powder or slurry form gave about equivalent results to quicklimes used in a slurry form.

A study indicated that a slaking time of ½ hour was sufficient and that any further slaking produced no beneficial results.

Chairman Goetz.—In this work were any tests made on the durability of these stabilized mixtures, or are all of these conclusions based upon unconfined compressive strength tests?

Mr. Sheeler.—We have done some previous research with lime stabilization of soil and with lime and fly ash mixtures and have come to the conclusion that an unconfined compressive strength of (I shall not mention a specific figure because I am not sure about this) somewhere around 400 or 500 psi was sufficient to pass freezing and thawing and wetting and drying tests satisfactorily.

In this research we did not do any freezing and thawing or wetting and drying testing.

Chairman Goetz.—I have another question. Do you have any explanation as to why dolomitic lime was much more effective in these soils than calcitic limes?

Mr. Sheeler.—This is a question that we have worked on, and we have more or less come to the conclusion that it is somewhat due to catalysis, the pozzolanic reaction being catalyzed by the presence of magnesium oxide in the dolomitic lime. But we know that this is not the exact answer because

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*Professor of Highway Engineering, Purdue University, Lafayette, Indiana.

we get a definite ratio of calcium to magnesium oxide, a molar ratio, that gives us the best results when using synthetic limes, that is mixtures of pure calcium oxide and magnesium oxide. This seems to indicate that magnesium oxide is entering the pozzolanic reaction, probably to form some crystalline or pseudocrystalline substance in which the magnesium oxide is an integral part of the crystalline structure of the pozzolanic cement.

Mr. Carl A. Carpenter.†—Has any attempt been made to explain or find out why the stability did not follow the density curves on these soils as they were tested for stability?

Mr. Sheeler.—This is due to the reaction between the soil and the lime which cements the soil and increases strength; the density decreases with increasing lime content because of flocculation.

Does that answer you satisfactorily?

Mr. Carpenter.—I would say the answer is that there was no direct study made of the density, and that probably there would have been very little point in making such a study of a pozzolanic mixture.

Mr. Sheeler.—No, the action of lime is like Portland cement. When lime reacts, it cements the soil together whether the density is high or low. The stability is mainly due to cementation. However, for a given soil and lime mixture, an increase in compacted density generally improves cementation and resulting stability.

Mr. Paul Phelan.‡—My question concerns the use of powdered lime versus lime slurry. It was mentioned that the use of lime slurry might be limited in some cases because of the optimum moisture requirement for the soil being stabilized with lime. Does this optimum moisture content change with the addition of lime? In other words, is the optimum moisture content for compaction of raw soil different than the optimum moisture content for compaction of the soil and lime mixture?

Mr. D. T. Davidson (author).—The optimum moisture content of soil is generally increased by admixtures of lime, the increase being greater for clayey soils than for silty soils. This is illustrated by the data in table III.

Mr. Kenneth A. Gutschick.§—I would like to pass on additional information on the use of quicklime in stabilization. Presently little or no dry quicklime is being used in this country. The Texas Highway Department experimented with it several years ago, but the workmen experienced some bad burns. This led the highway department to specify hydrated lime only.

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†Bureau of Public Roads, Division of Physical Research, Washington, D. C.

‡Technical Director, Road Materials, Koppers Co., Inc., Tar Products Division, Pittsburgh, Pennsylvania.

§Manager, Technical Service, National Lime Assn., Washington, D. C.

However, it should be pointed out that from a stabilizing standpoint, the job was entirely successful. The quicklime especially helped to dry up the wet clay subgrade.

The Corps of Engineers at Vicksburg has made some field experiments with quicklime for effecting quick stabilization in conjunction with moving military vehicles over wet subgrades. This work, described in a Corps bulletin, reported considerable success with the soil used.

R. S. Boynton, National Lime Assn. general manager, who recently returned from a trip to Germany, reports that considerable pulverized quicklime is being used in Germany in subgrade stabilization, particularly in wet cuts. Some of this work has been on Autobahn projects. Evidently the Germans have learned to handle the quicklime safely.

# LIME FIXATION IN CLAYEY SOILS

by

G. H. Hilt, Captain, Corps of Engineers, U. S. Army
D. T. Davidson, Professor, Civil Engineering

(Highway Research Board Bulletin 262:20-32. 1960.)

## INTRODUCTION

Desirable results obtained from the use of lime and lime fly ash additives to improve the engineering properties of clayey soils include better workability, increased immersed and dry strengths, increased resistance to freeze-thaw cycles, and better volume change characteristics.

Previous research has indicated that the addition of a very small percentage of lime improved the workability of heavy clay soils many fold but added little to strength. Additional lime improved the strength and bearing capacities of these soils, and the addition of fly ash caused an even more significant strength gain in some clayey soils.

### Scope and Objectives

That lime added to soil must first satisfy an affinity of the soil for lime was suggested by previous research. If this is true then it would be suspected that lime would not be available for the pozzolanic reactions with the soil constituents or fly ash needed to produce strength gains until the affinity of soil for lime is satisfied. Since this lime is *fixed* in the soil and is not available for other reactions, the process by which the lime becomes fixed may be termed *lime fixation.* The percentage amount of calcium hydroxide by oven dry weight of the soil which can be fixed by a given soil may be identified as the *lime fixation capacity* of the soil.

### Methods

If there is lime fixation in a clayey soil, the strength of compacted and cured specimens of the soil should not increase with small additions of lime. To confirm this unconfined compressive strength tests were used, since unconfined compression is a reliable indication of cohesive and cementing strength. This strength test can be performed relatively rapidly on a large number of specimens.

Since the liquid and plastic limits of a soil are affected by cohesiveness, one or both of these tests were investigated to determine if they also can be used as parameters of lime fixation capacity.

## REVIEW

Much has been written of the desirable effects of adding lime to a soil rich in clay. The addition of lime to over-wet clayey soils appears to dry them out and materially improve their workability[14]. In highly plastic soils in Texas the ease of pulverizing clay balls after lime and water was added exceeded expectations, and during final mixing and placing with motor patrols the material was very friable and had many characteristics of a non-plastic mix[8]. Reductions in the plasticity index with additions of less than three percent lime were noted in both of the above.

Increases in strength with the addition of lime have been observed by a great many researchers. The Iowa State University Engineering Experiment Station Soil Research Laboratory has been carrying out research projects on the treatment of soils with lime and with lime and fly ash since 1953. Publications on the research show favorable results of increasing strength of soils with the addition of lime and of lime and fly ash[12].

Some of the basic mechanisms involved have been explained[7]. First, calcium ions cause a reduction in plasticity of cohesive soils so they become more friable and more easily worked. The mechanism is either a cation exchange or a crowding of additional cations onto the clay. Both processes change the electrical charge density around the clay particles. Clay particles then become electrically attracted to one another, causing flocculation or aggregation. The clay particles, now acting as aggregates, behave as a silt which has a low plasticity or cohesion. A second chemical reaction is carbonation of lime by carbon dioxide of the air, producing calcium carbonate, a weak cement which is deleterious to overall strength gains. A third class of reactions, termed pozzolanic reactions, results in a slower, long-term cementation of compacted mixtures of lime and soil. Pozzolanic reactions apparently involve interactions between hydrated lime and minerals in the soil.

Lime contents of 1% and less in a clayey soil are reported to produce a metastable state; but when lime contents greater than that are used, the flocculation of the clay particles is of a more permanent and progressive nature[5]. Calcium silicate and aluminate formed by chemical breakdown of the clay lattice material contribute to flocculation by bonding adjacent soil particles. Ionic flocculation and silicate bonding commence at the same time, the former being an immediate effect while the latter takes a considerable time to complete. Nearly all of the clays used were already saturated with adsorbed calcium; changes in properties as a result of additions of calcium hydroxide could not involve a cation exchange relationship.

An addition of 8% calcium hydroxide to clayey soils resulted in the complete conversion of $Ca(OH)_2$ to other forms after one month[10, 13]. Reactions other than ion exchange probably accounted for the conversion. However, in other research calcium hydroxide was found to be still present after twelve months curing of specimens[5].

Changes in the engineering properties of clayey soils with addition of lime depend appreciably on the cation originally adsorbed on the clay surfaces, and on the type of clay[11]. The nature of the exchangeable cation does not make much difference in kaolinitic soils, but it makes a tremendous difference in montmorillonitic soils.

Expanding clays containing montmorillonite react readily with lime immediately losing plasticity, and after compaction slowly gain pozzolanic strength. Clays containing mainly illite, chlorite, vermiculite, or kaolinite are less effective users of lime[7].

Laboratory tests on two soils of high clay content indicate that no appreciable pozzolanic reactions can take place between lime and fly ash until lime is present in excess of the requirements of the soil[6].

## SPECIMEN PREPARATION AND TESTING

### Materials

*Soils.* Seven soils were used in the investigation. The choice of these soils was based on the type and amount of the principal clay minerals present.

TABLE I. SOIL SITE CHARACTERISTICS

| Sample | Location | Classification | Soil series and horizon | Sampling depth in inches |
|---|---|---|---|---|
| M-67 | Keokuk County, Iowa | Kansan-age Gumbotil | Mahaska,* fossil B horizon | 91-101 |
| M-51 | Harris County, Texas | Coastal plain deposit, largely deltaic | Lake Charles, probably B horizon | 39-144 |
| M-33 | Keokuk County, Iowa | Plastic Wisconsin-age loess | Mahaska, C horizon | 36-77 |
| I-44 | Monroe County, Michigan | Probably Wisconsin-age glacial till | Unknown, probably C horizon | Unknown |
| I-41 | Livingston County, Illinois | Wisconsin-age glacial till | Clarence, C horizon | 46-56 |
| K-30 | Durham County, North Carolina | Residual soil over medium grained biotite granite | Durham, B horizon | 24" below A horizon |
| K-29.5 | Orange County, Virginia | Residual soil over diorite | Davidson, B horizon | Unknown |

*Under soil M-33

Three soils containing montmorillonite, two containing illite-chlorite mixtures, and two of the kaolinite group (one containing kaolinite, and the other halloysite) were selected. Each of the soils is identified by a letter designating the principal clay minerals present, and a number indicating the percentage content of the soil less than two microns in diameter. For example, the designation, M-75, indicates a soil containing montmorillonite as the principal clay mineral, with 75% of this soil less than $2\mu$ in effective diameter. The locations from which the soil samples were taken and other pertinent information appear in table I. Table II gives the physical and chemical properties of the seven soils.

TABLE II. PROPERTIES OF SOILS

| Sample | M-67 | M-51 | M-35 | I-44 | I-41 | K-30 | K-29.5 |
|---|---|---|---|---|---|---|---|
| I.E.E.S. designation | 528-8 | AR-3 | 528-4 | AR-4 | AR-8 | AR-6 | AR-5 |
| Textural composition | | | | | | | |
| Gravel* > 2 mm. | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Sand (2—0.074 mm.) | 16.0 | 3.0 | 0.2 | 7.0 | 10.0 | 45.2 | 21.0 |
| Silt (74—5$\mu$) | 15.5 | 36.0 | 60.8 | 36.0 | 38.0 | 18.3 | 37.0 |
| Clay (5$\mu$) | 70.5 | 61.0 | 39.0 | 57.0 | 52.0 | 36.5 | 42.0 |
| Clay (2$\mu$) | 67.0 | 51.0 | 33.0 | 44.0 | 41.0 | 30.0 | 29.5 |
| Passing No. 10 sieve | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Passing No. 40 sieve | 96.0 | 99.0 | 100.0 | 98.0 | 96.0 | 67.0 | 90.0 |
| Physical properties | | | | | | | |
| Liquid limit, % | 76.6 | 64.6 | 52.1 | 44.0 | 35.5 | 51.0 | 43.5 |
| Plastic limit, % | 25.6 | 17.6 | 20.0 | 21.1 | 17.5 | 25.5 | 27.0 |
| Plasticity index | 50.0 | 47.0 | 32.1 | 22.9 | 18.0 | 25.5 | 16.5 |
| Chemical properties | | | | | | | |
| pH | 7.1 | 8.2 | 5.6 | 8.4 | | 5.7 | 5.9 |
| C.E.C. (soil passing No. 10 sieve), m.e./100 g | | 27.5 | 23.5 | 14.5 | | 8.4 | 11.0 |
| C.E.C. (soil passing No. 40 sieve), m.e./100 g | 41.0 | 33.1 | 26.8 | 13.4 | | 13.5 | 12.4 |
| Carbonates, % | 0.8 | 16.6 | 0.0 | 7.2 | | 0.1 | 0.7 |
| Organic matter, % | 0.2 | 0.1 | 0.2 | 0.6 | | 0.1 | 2.6 |
| Predominant clay | | | | | | | |
| Classification mineral† | M | M | M | I & C | I & C | K | H & V |
| Textural‡ | Clay | Clay | Silty clay | Clay | Clay | Clay | Clay |
| Unified | CH | CH | CH | CL | CL | CH-CL | CL-ML |
| BPR (AASHO) | A-7-6(20) | A-7-6(20) | A-7-6(18) | A-6-6(14) | A-6 (11) | A-7-6(11) | A-7-6(12) |

*Textural gradation tests were performed only on the soil fraction passing the No. 10 sieve. All soils used contained less than 5% gravel.
†Symbols are M—montmorillonite, I—illite, C—chlorite, K—kaolonite, H—halloysite, and V—vermiculite. Determinations were made by X-ray diffraction.
‡U. S. Dept. of Ag. textural classification was used.

*Lime.* Reagent grade calcitic hydrated lime, $Ca(OH)_2$, was used to minimize compositional variables. Individual one pound bottles of lime were kept sealed until immediately before use to prevent carbonation of the lime.
*Fly Ash.* Fly ash is "the finely divided residue that results from the combus-

tion of ground or powdered coal and is transported from the boiler by flue gas"[4]. The fly ash used was collected at the St. Clair Power Plant of the Detroit Edison Company, Detroit, Michigan[2]. This fly ash had a loss-on-ignition 3.6%, and 88.7% of the ash was finer than the No. 325 sieve.

## MIXTURE AND SPECIMEN PREPARATION

### Soil preparation

The soil to be used was selected from the appropriate bin and was run through the pulverizer to reduce all soil aggregations to particle size or fine enough to sieve through either the No. 10 or the No. 40 mesh sieve, depending on whether the soil was to be used for strength tests or for liquid and plastic limits tests. The soil passing the appropriate mesh sieve was then put through a sample splitter, placed in covered cardboard containers, and stored until needed.

### Mixing

A predetermined amount of air dry soil was weighed on a balance sensitive to 0.1 gram and then placed in a mixing bowl. Additives, if used, were weighed and mixed in at low speed with a mechanical mixer. After mixing the soil and additives together dry for thirty seconds, distilled water was added in appropriate amounts, and mixing was continued for another four minutes.

### Unconfined Compression Test Specimen Preparation

Strength test specimens were 2 by 2 inches and were molded in an apparatus developed at the Iowa State University Engineering Experiment Station Soil Research Laboratory. The apparatus (figure 1) is a hand operated drop hammer with which a predetermined amount of soil mixture in a 2 inch diameter mold is compacted to a density near standard Proctor density.

Approximately 200 grams of the mixture of soil, additives, and water is placed in the mold by means of a scoop and funnel. This mixture is then given five blows from the hammer, the mold is inverted, and another five blows are applied. The resulting soil cylinder is extruded from the mold with an hydraulic jack. The compacted specimen is weighed to the nearest 0.1 gram, and the height is measured to the nearest 0.001 inch. Any specimen not measuring 2.0 inches $\pm$ 0.05 inch is rejected. The soil specimen is then wrapped in waxed paper and sealed with cellulose tape to prevent carbonation of the lime by the carbon dioxide in the air. The wrapped specimens are placed in shelves in a curing room where the relative humidity is maintained at 95 $\pm$ 5% and the temperature at 70 $\pm$ 5° F.

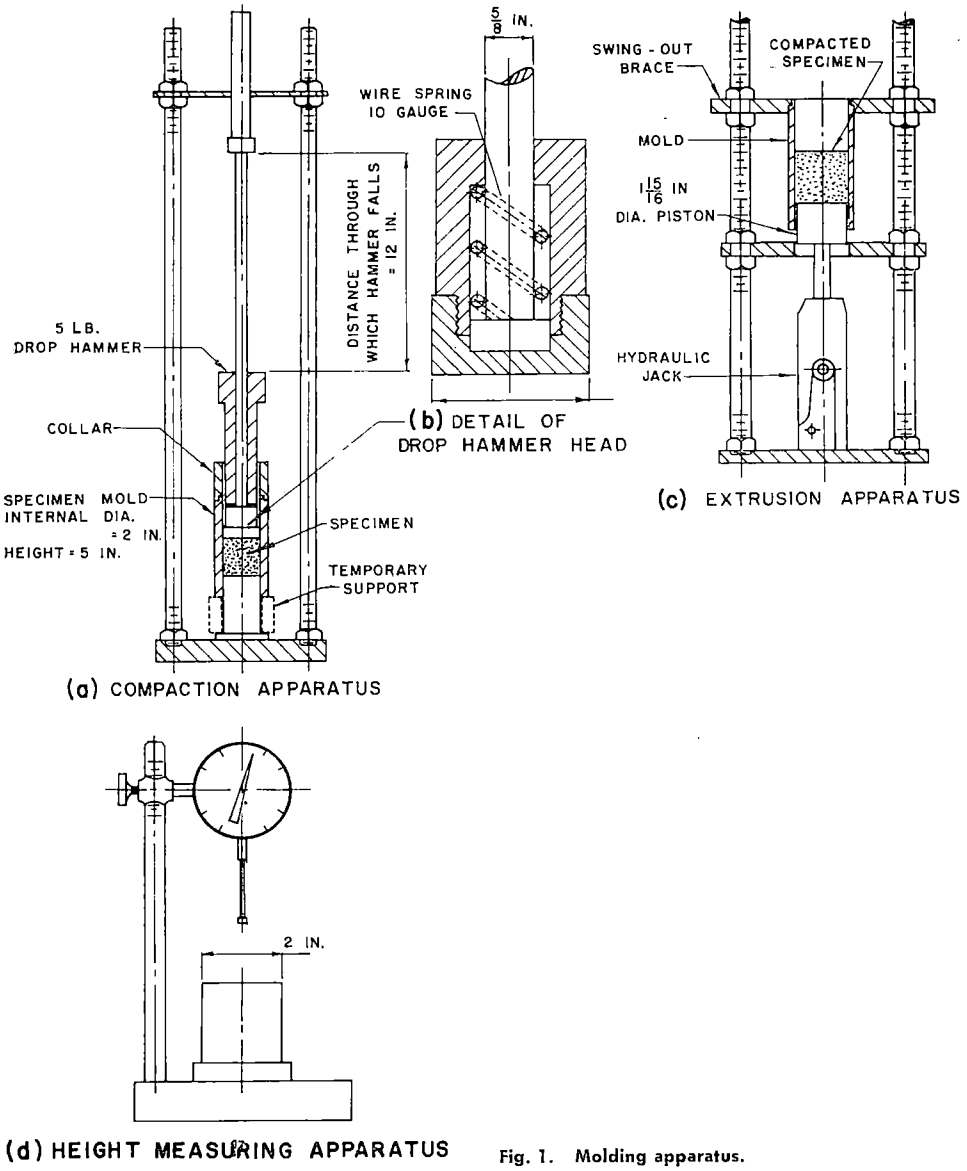


Fig. 1. Molding apparatus.

## Testing

*Liquid and Plastic Limits.* ASTM Methods D423-54T and D424-54T[1] were followed except that after the soil, lime, and distilled water were mixed together, the mix was scraped into a porcelain pan, covered, and stored in a near 100% humidity room for two days. This allowed the water to infiltrate the clay particles thoroughly and produce uniform wetting of the

sample. Preliminary tests after different storage periods up to four days showed no perceptible daily change in the liquid and plastic limits after two days storage.

After seasoning for two days the contents of the covered pan were removed from the humidity room, and enough of the mixture was placed in an evaporating dish for four liquid limit tests. This procedure was followed on the soil passing the number 40 mesh sieve with additives of 0, 1, 2, 3, 4, 7, and 12% lime by oven dry weight of soil (hereafter abbreviated dry weight of soil). Liquid limits were determined only for mixtures prepared with the montmorillonitic and illitic, chloritic soils. Because of the wide variety of variables which enter into a liquid limit test, consistency, and reproducibility of results were difficult to obtain. Since the plastic limit was found to produce reliable and consistent results correlating well with the data to be presented, liquid limit tests were discontinued.

A sample weighing about eight grams was taken from the mixture prepared for the liquid limit test. Four plastic limits were rolled for each soil lime mixture studied (0, 1, 2, 3, 7, and 12% additions of lime by dry weight of soil). The plastic limit of the mix at each percentage additive was determined as the average of the moisture contents of the four threads rolled.

**Strength Tests**

*Apparatus.* The testing apparatus was a model AP-170 Stability Testing Machine manufactured by Soil Test, Inc. Loads are indicated on a 10,000 pound capacity proving ring, which has a dial indicator reading to 0.0001 inch deflection. Strain is applied to the test specimens at a constant rate of 0.1 inch per minute.

*Preliminary Studies.* Since the objective of the investigation was to study the process of lime fixation in clayey soils, it was important to obtain positive values of strength for all mixtures of soils and additives. Therefore, strength testing was done on specimens which had been moist cured but not subjected to immersion, since immersion causes specimens of low lime content to slake.

To obtain the optimum moisture content for maximum strength for molding the 2 by 2 inch specimens, moisture-strength tests were conducted in series with four sets of three specimens each, molded for each soil mix at varying moisture contents. After seven days moist curing (95 $\pm$ 5% R. H., 70 $\pm$ 5° F) these specimens were tested for unconfined compressive strength, and the strengths of the specimens at failure were recorded. Graphs of molding moisture content versus seven day strength were prepared for each mixture studied, and optimum moisture contents for maximum strengths were interpolated. This procedure was carried out for additives of 0, 6, and 12% lime for each soil and also for the same percentages of lime plus 20% fly ash. The optimum moisture contents thus obtained

were then plotted against lime contents for each soil. The resulting curves were used for interpolating moisture contents for molding specimens of the various mixtures used in the final work.

*Final Testing.* The soils with the highest percentage of each type of clay mineral (M-67, IC-44, and K-30) were selected for final testing. Addi-

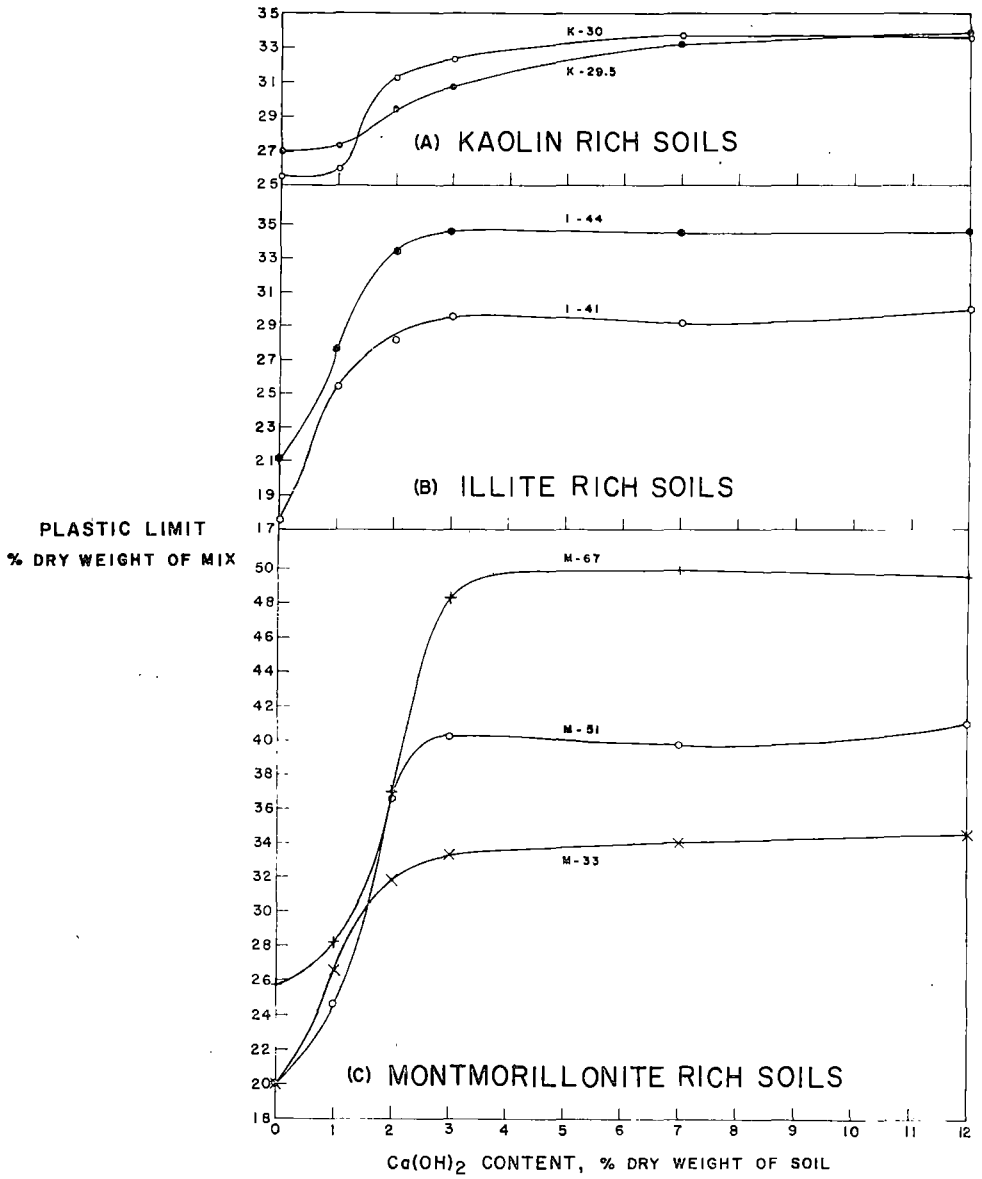


**Fig. 2. Plastic limits of seven clayey soils at varying lime contents.**

tives to the soils fell into two groups: the first was lime and the second, lime and fly ash. Lime alone was added to each of the soils in amounts of 0, 1, 2, 3, 4, 6, 8, and 12% by dry weight of soil. In the second group the same percentages of lime by dry weight of soil were used, but 20% fly ash by dry weight of soil also was added to provide an excess of pozzolanic material.

Batches for molding nine specimens out of each were prepared to obtain uniformity of mixing. All batches of each group for a soil were molded on the same day to assure uniform curing conditions. Of the nine specimens molded, three were tested at the end of seven days curing in the humidity room, three at 28 days, and the final three after 27 days moist curing and one day immersion in distilled water.

## RESULTS

### Effect of Lime on the Plastic Limit

The plastic limits of all soils and mixtures tested showed that this limit was materially increased with small additions of lime (figure 2). The largest increases in the plastic limit (P. L.) due to lime treatment were obtained in the soils containing montmorillonite as the principal clay mineral; the greater amount of clay-size material in these soils, the greater was the increase in the P. L. Increases in the P. L. of the illitic-chloritic-clayey soils also were considerable, but not as great as in the montmorillonitic soils of comparable clay-size content. The smallest increases of the P. L. were observed in the kaolinitic rich soils.

Of particular interest is the "point" on each curve at which the rate of change of the slope approaches zero and at which the slope itself approaches zero. Since the curve approximates that of a right hyperbola, the change in slope never reaches zero but approaches this value in soils M-67, M-51 and M-33 at lime contents of 3.2%, 2.7% and 2.2%, respectively. A plot of these lime percentages against respective 2 micron clay contents (table II) of the soils revealed the following linear relationship:

$$\text{Optimum lime additive for maximum increase in plastic limit} = \frac{\%\text{ of } 2\mu \text{ clay}}{35} + 1.25 \qquad (1)$$

Since only two illitic-chloritic soils and two kaolinitic soils were tested, it is not known whether or not the optimum lime additive for these two soils is directly proportional to 2 micron clay content.

To determine the factors responsible for the observed change in P. L. with the addition of lime, the definition of the plastic limit must be stated in terms of the events occurring in the laboratory test. The definition of P. L. (1) as the boundary between the plastic and semi-solid states must be modified to a definition that reflects laboratory procedure. Thus a more

descriptive definition of the P. L. as determined by the laboratory soil test is the lowest moisture content at which the bonds between soil particles or aggregates can be constantly renewed.

The nature of these bonds has been described as being due to cation exchange and to a crowding of additional cations onto the surfaces of the clay[7]. However, one of the soils tested, M-51, already had an excess of carbonates present (16.6%). Certainly, then, this soil is already saturated with calcium, yet the P. L. of the soil was increased from 18 to 40 with the addition of less than 3% lime. Thus the crowding of additional calcium onto the clay must be the more important of the two mechanisms.

One other mechanism not previously mentioned enters into the bonding of clay particles and affects the plastic limit. The surface tension of water in minute pores such as those in clayey soils exerts a bonding force between clay size particles. Since capillary pressure is inversely proportional to the radius of curvature of the moniscus and directly proportional to surface tension, changes in either of these will be reflected in the amount of water necessary to renew the bonds continually between soil particles while a plastic limit is being rolled.

When the plastic limits of the soils tested are increased by lime additives, actually more water must be added to the soil and lime mixture to make the bonds between the soil particles capable of being renewed as rapidly as they are broken (figure 2). The addition of lime causes the clay particles to become electrically attracted, causing flocculation or aggregation. The clay then behaves more like a silt with each floc of clay particles acting like a silt grain. Though bonding between the particles within a floc has been increased, bonding between flocs is relatively weak. When a plastic limit is rolled, the bonds between individual clay particles within the floc remain relatively unaffected, but the bonds between the flocs are being constantly broken and reformed until the moisture content is reduced to the point where the bonds can no longer be reformed as fast as they are being broken, and the thread crumbles.

At some percentage of lime additive all of the calcium that can crowd onto the clay particles is present and further increases in lime result only in supplying to the soil an excess of calcium which is not effective in flocculation or other mechanisms which contribute to the increase in the plastic limit.

If the calcium which increases the plastic limit is so fixed in the soil that it cannot react with the natural pozzolans of the soil, then the percentage of lime present at the point where the plastic limit reaches its maximum will reliably indicate the percentage of lime fixation in the soil.

**Effect of Lime and of Lime and Fly Ash on Strength**

*Unconfined Compressive Strengths.* The three soils with the greatest

amount of each clay mineral, M-67, IC-44, and K-30, were selected for determining the effect of lime and lime fly ash treatments on the unconfined compressive strengths of clayey soils (figures 3, 4, 5).

The addition of lime increased the unconfined compressive strength of montmorillonitic soil M-67 the largest amount. The strength of kaolinitic soil K-30 also was greatly improved by lime treatment but the strength

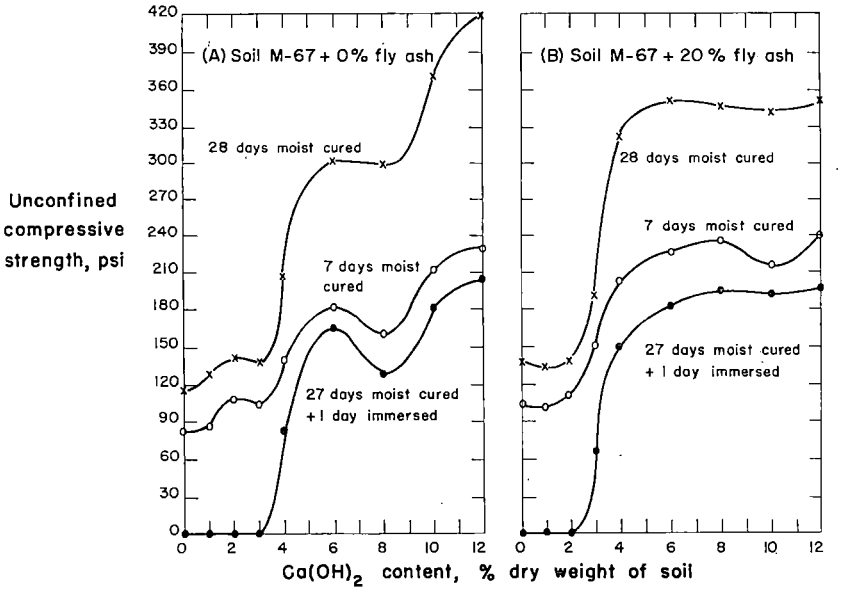


**Fig. 3. Unconfined compressive strengths of soil M-67 at varying lime and lime fly ash contents.**

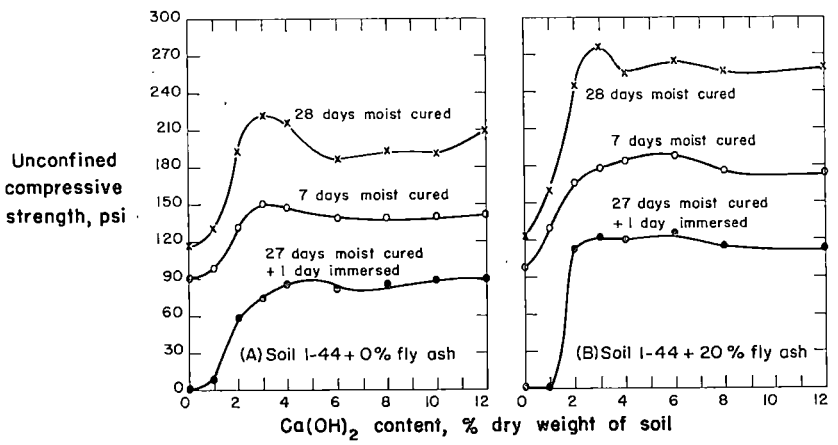


**Fig. 4. Unconfined compressive strengths of soil I-44 at varying lime and lime fly ash ratios.**

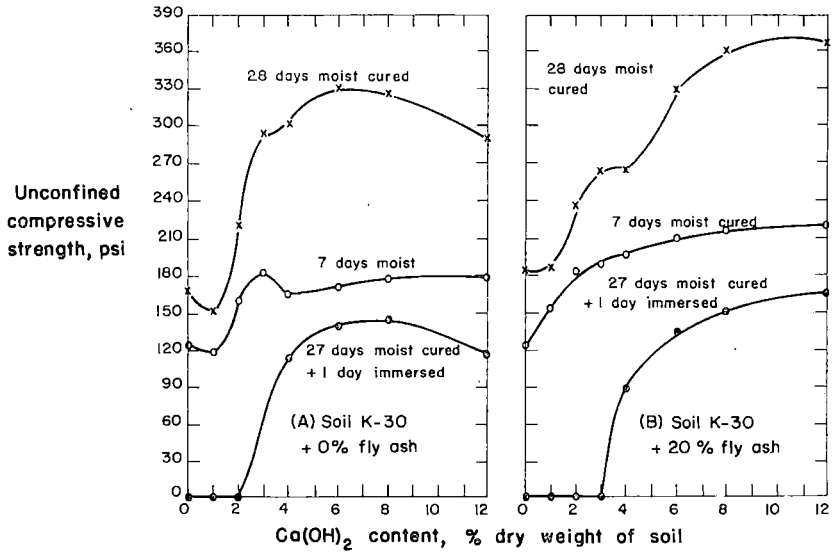


**Fig. 5. Unconfined compressive strengths of soil K-30 at varying lime and lime fly ash ratios.**

of illitic-chloritic soil IC-44 was only slightly increased.

The addition of 20 percent fly ash to the soil plus lime had little effect on soils M-67 and K-30, but produced marked gains in unconfined compressive strength in soil I-44. A comparison of the maximum unconfined compressive strengths of soil IC-44 plus lime after 28 days moist curing with and without fly ash additive shows that the addition of 20 percent fly ash has increased the strength of this soil by 28 percent.

From this data it appears that both montmorillonite and kaolinite are natural pozzolans; and they, or elements within their crystal lattices, react with lime to produce cementing materials. Since soil M-67 contains much more minus two micron material than soil K-30, it can be expected that lime mixed with soil M-67 will produce more cementing materials than will lime and soil K-30 even if both clays are equally reactive.

However, since the addition of lime to soil IC-44 produces only relatively small increases in unconfined compressive strengths, one can conclude that either illite or chlorite or both are not effective natural pozzolans. Thus it is necessary to add a pozzolanic material such as fly ash to obtain significant increases in strength in stabilization with lime.

*Proof of Lime Fixation.* The initial and generally flat portion of the unfined compressive strength curves is of particular interest. In this region small additions of lime do not produce corresponding increases in strength. This shows graphically that a certain amount of lime must be added to a clayey soil before cementing products which will increase the strength of

the soil can be formed. Since lime fixation was defined as the process by which lime is held by the soil and is not available for pozzolanic reactions, this initial flat portion of the curves proves that lime fixation does occur in clayey soils.

The region of lime fixation may be defined as that portion of the unconfined compressive strength versus lime content curves in which strength does not perceptibly increase as the percentage of lime additive increases. Similarly, the lime fixation capacity of a soil is the percentage of lime additive at which strength just begins to increase.

**Comparison of Plastic Limits and Strengths**

*Object of Comparison.* By comparing the optimum lime additive for maximum increase in the plastic limit (figure 2 and equation 1) with the maximum percentage of lime fixation which can occur in a particular soil (figures 3, 4, 5) it can be shown that these two percentages are the same, and thus this optimum on the plastic limit curves is also a quantitative parameter of the lime fixation capacity of a particular soil.

*Method of Comparison.* If the unconfined compressive strengths of the soils tested remain constant as the plastic limits increase with increasing percentages of lime additive, and if the plastic limit then remains constant with further additions of lime as the unconfined compressive strengths increase, then the optimum lime additive for maximum increase in the plastic limit will be a quantitative parameter of lime fixation (figure 6).

Since previous research has shown that lime does not react with the

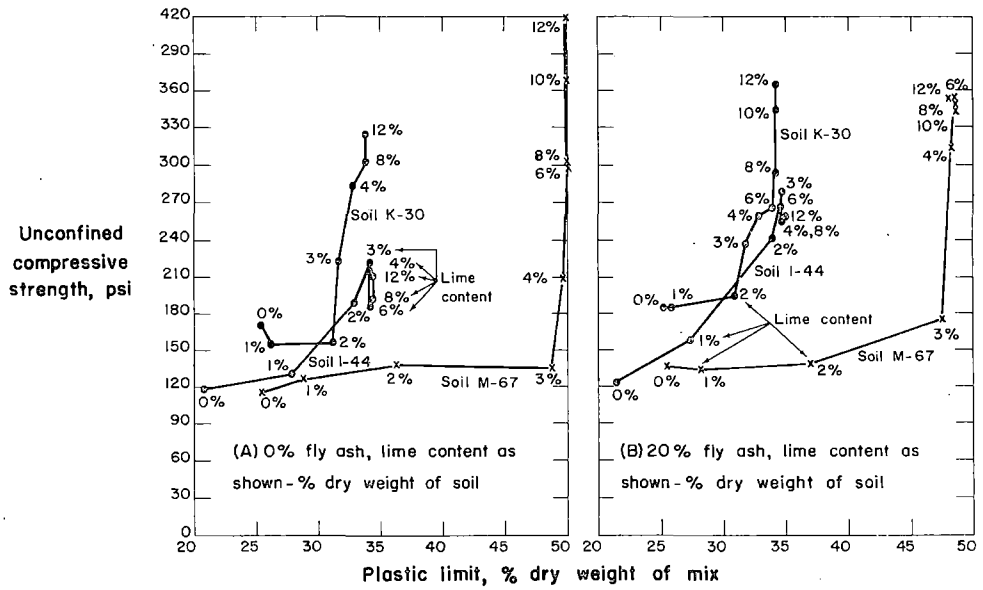


**Fig. 6. Comparison of plastic limits and unconfined compressive strengths for three lime-treated clayey soils.**

coarse portion of the soil and since P. L. tests utilized the portion of the soil passing the No. 40 sieve and unconfined compressive strength testing was performed on cylinders molded from the soil passing the No. 10 sieve, a correction must be made[15]. This correction is based on relating the percentage of lime additive to the amount of clay present in the soil passing the No. 10 sieve. Thus the equivalent percentage of lime in the plastic limit tests is

$$L_E = L_o \times \frac{Q_{40}}{Q_{10}} \qquad (2)$$

in which $L_E$ = equivalent lime content (%)
$L_o$ = original lime added (%)
$Q_{40}$ = amount of soil passing the No. 40 sieve (%)
$Q_{10}$ = amount of soil passing the No. 10 sieve (%)

*Results and Discussion.* The curves (figure 6) show that the compressive strengths of soils M-67 and K-30 remain constant as the plastic limits increase, and the plastic limits remain constant as the strengths increase.

However, in soil IC-44, which contains illite and chlorite as the principal clay minerals, the strength is increasing while the plastic limit is still increasing. A possible explanation for this is that if one of the two clay mineral constituents of the soil has a lower lime fixation capacity than the other, then, at a percent additive of lime at which one is still engaged in the process of lime fixation, the other may have completed this process and begun to engage in the production of cementing materials through pozzolanic reactions.

*Conclusions from Comparison.* Based on the graphical results of this comparison the optimum lime additive for maximum increase in the plastic limit is a reliable quantitative indicator of the lime fixation capacity of the montmorillonitic and kaolinitic soils tested. However, it does not reliably indicate the lime fixation capacity of clayey soils containing a mixture of illite and chlorite, though it may be a valid indicator in soils containing one or the other of these minerals.

## CONCLUSIONS

1. Large increases in the plastic limits of clayey soils can be obtained by adding small quantities of lime, $Ca(OH)_2$. The largest increases are in soils containing montmorillonite; illitic-chloritic clayey soils are affected somewhat less, and the plastic limits of kaolinitic clayey soils are the least changed.

2. A descriptive definition of the plastic limit as determined by the standard laboratory method is the lowest moisture content at which the bonds between soil particles or aggregations can be constantly renewed.

3. Both montmorillonite and kaolinite are effective pozzolanic reagents. They or elements within their crystal structure will react with lime

to produce a cementing material equal to or greater in strength producing qualities than the cementing agents produced in the reaction of lime with fly ash. It is not necessary to add fly ash to soil containing large amounts of montmorillonite or kaolinite when treating with lime. Such additions may even be detrimental.

4. The percentage amount by oven dry weight of the soil of calcium hydroxide which can be fixed by a given soil is the lime fixation capacity of that soil. This lime contributes to the improvement of soil workability but not to increases in strength. Amounts of lime added above the lime fixation capacity cause the formation of cementing materials within clayey soils.

5. Lime fixation in clayey soils does take place. The lime fixation capacity of a montmorillonitic or kaolinitic clayey soil is the same as the optimum lime additive for maximum increase in the plastic limit of the soil.

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# IMPROVEMENT OF LIME STABILIZATION OF MONTMORILLONITIC CLAY SOILS WITH CHEMICAL ADDITIVES

by

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## ABSTRACT

The findings and interpretations of an investigation of the effects of additives of sodium phosphate, sodium carbonate or sodium hydroxide on the immersed strength of compacted mixtures of montmorillonitic clay soils and lime are presented. Also studied were the effects of curing temperature on the immersed strength and the durability of selected soil, lime and soil, lime, and chemical mixtures.

## INTRODUCTION

Research and field experience have shown that lime treatments of clayey soils produce beneficial property changes, but that the strength gain of compacted mixtures may be too slow to meet base course requirements, particularly in the northern United States where desirably high curing temperatures can not be counted on in late season construction, and where alternate cycles of freeze and thaw may cause failures of insufficiently hardened lime stabilized clayey soils. The results of recent work with chemical additives to improve the strength of soil cement and soil, lime, and fly ash[2, 3, 4, 6, 7, 11] suggested that perhaps chemical additives could be used to improve the cement producing (pozzolanic) reactions in clayey soil lime mixtures, giving rise to greater early strengths up to 28 days.

Heat is another possible way to accelerate the hardening of lime treated soils[5]. While it may not be economical at present to cure a soil, lime base course with artificial heat, future cheap sources of heat may make this feasible. The effect of curing temperature on the rate of strength gain of compacted soil and lime mixtures is of practical interest in the natural temperature range attained under field conditions.

This paper presents the results of a laboratory investigation of the above mentioned methods of improving the strength and durability of compacted clayey soil and lime mixtures. Stability requirements for road bases

in Iowa are used as the basis for evaluating the need for and the adequacy of the improvements.

## MATERIALS

### Properties of soils

Three montmorillonitic clay soils were selected for the investigation (tables I, II). The soils will be referred to as Kansan till, plastic loess and Kansan gumbotil.

These three are typical of subgrade soils found in southern Iowa and parts of adjacent states, where, due to a lack of local deposits of granular road materials, stabilization methods for clayey soils are badly needed.

TABLE I. BRIEF DESCRIPTION OF SOILS

| Soil | Kansan till (409-12C)* | Plastic loess (528-4) | Kansan gumbotil (528-8) |
|---|---|---|---|
| Location: | Ringgold County, Iowa | Keokuk County, Iowa | Keokuk County, Iowa |
| Geological description: | Kansan-age glacial till, plastic, calcareous, oxidized | Wisconsin-age loess, plastic, noncalcareous | Kansan-age gumbotil, highly weathered, plastic, noncalcareous |
| Soil series: | Shelby (Burchard) | Mahaska | Mahaska† |
| Horizon: | C | C | Fossil B |
| Sampling depth: | 4½ - 10½ ft. | 3 - 6½ ft. | 7½ - 8½ ft. |

*Numbers in parentheses are those assigned by the Soil Research Laboratory of the Iowa Engineering Experiment Station.

†Underlies C horizon loess of Mahaska series.

Kansan till and plastic loess contain approximately the same amount of montmorillonitic clay; but the non-clay size fraction of the two soils is considerably different. In Kansan till this fraction is about one-half silt sizes and one-half sand sizes; in plastic loess it is all silt sizes. Another difference is in carbonate content; Kansan till is calcareous, and plastic loess is non-calcareous or leached. The clay particles in a calcareous soil should be in a flocculated state with their appetite for calcium ions mostly satisfied, and nearly all added lime is available for pozzolanic reactions. In a leached soil, initial additions of lime furnish the calcium ions needed to flocculate the clay particles. Thus less lime should be required to stabilize Kansan till than plastic loess.

Kansan gumbotil is a unique soil type. Supposedly a fossil B horizon developed from Kansan till, this highly weathered noncalcareous soil contains about 70% montmorillonitic clay, almost twice as much as the other

two soils (table II). The non-clay fraction of Kansan gumbotil is about equal parts sand and silt-size material.

TABLE II. PROPERTIES OF SOILS

| Soil | Kansan till | Plastic loess | Kansan gumbotil |
|---|---|---|---|
| Textural composition,* % | | | |
| Gravel (> 2 mm) | 0.0 | 0.0 | 0.0 |
| Sand (2.0 - 0.074 mm) | 31.5 | 0.2 | 16.0 |
| Silt (0.074 - 0.005 mm) | 30.0 | 60.8 | 13.5 |
| Clay (< 0.005 mm) | 38.5 | 39.0 | 70.5 |
| Clay (< 0.002 mm) | 31.0 | 33.0 | 67.0 |
| Atterberg limits†: | | | |
| Liquid limit, % | 42.4 | 52.1 | 75.6 |
| Plastic limit, % | 20.5 | 20.0 | 25.6 |
| Plasticity index, % | 21.9 | 32.1 | 50.0 |
| Classification: | | | |
| Textural‡ | Clay | Silty clay | Clay |
| Engineering (AASHO)§ | A-7-6(12) | A-7-6(18) | A-7-6(20) |
| Chemical: | | | |
| Cat. exch. cap.,¶ m.e./100 g | 29.5 | 23.5 | 41.0 |
| pH£ | 8.25 | 5.6 | 7.1 |
| Carbonates,** % | 4.9 | 0.0 | 0.8 |
| Organic matter,†† % | 0.17 | 0.2 | 0.2 |
| Predominant clay mineral‡‡ | Montmorillonite | Montmorillonite | Montmorillonite |

* ASTM Method D 422-54T (8).
† ASTM Method D 423-54T and D 424-54T (8).
‡ Triangular chart developed by U. S. Bureau of Public Roads (9).
§ AASHO Method M 145-49 (10).
¶ Ammonium acetate (pH = 7) method of soil fraction < 0.42 mm (No. 40 sieve).
£ Glass electrode method using suspension of 15 g soil in 30 cc distilled water.
**Versenate method for total calcium.
††Potassium bichromate method.
‡‡X-ray diffraction analysis.

### Properties of limes

Both types of lime, the dolomitic and the calcitic, were commercial grade hydrated limes produced by plants of the U. S. Gypsum Company, Chicago, Illinois (table III).

### Properties of chemicals

Three chemical additives were used, chosen as among the more promising for the improvement of pozzolanic reactions[2]. The sodium carbonate ($Na_2CO_3$) was produced by the J. T. Baker Chemical Company, catalog No. 3602; the sodium phosphate ($Na_3PO_4 \cdot 12H_2O$) by the Mallinckrodt Chemical Works, catalog No. 7940; and the sodium hydroxide (NaOH) by the Mallinckrodt Chemical Works, catalog No. 7708. All three chemicals were analytical reagent grade.

TABLE III. PROPERTIES OF LIMES

| | Dolomitic (Monohydrate, Type N) | Hydrated limes Calcitic (Hi-calcium) |
|---|---|---|
| Chemical analysis, percent by weight: | | |
| Silicon dioxide | 0.4 | 0.28 |
| Iron and aluminum oxide | 0.3 | 0.6 |
| Magnesium oxide | 31.8 | 0.59 |
| Sulfur trioxide | 1.1 | 0.25 |
| Carbon dioxide | 1.0 | ND* |
| Total calcium oxide | 48.8 | 73.82 |
| Available calcium oxide | 47.1 | 70.3 |
| Loss on ignition | 17.0 | 24.1 |
| Combined $H_2O$ | 18.0 | ND* |
| Processing location: | Genoa, Ohio | New Braunfels, Texas |
| Trade name: | Kemidol Hydrated | Kemikal Hydrated |

*Not determined.

## METHODS

### Preparation of mixtures

Air dried soil passing the No. 10 sieve was dry mixed with the lime additive for thirty seconds by a Hobart Model C-100 mixer. Sufficient water was then added to bring the moisture to the desired optimum content and mixing was continued for two minutes. The mixture was next hand mixed a few times to loosen any of the soil stuck to the sides of the mixing bowl. It was then machine mixed for another two minutes. Chemical additives, when used, were dissolved in the mix water prior to adding to the soil, lime mixture. The weight of lime or chemical additive used was expressed as a percentage of the oven dry weight of the soil. The percentages of lime, sodium carbonate or sodium hydroxide refer to the as-received chemical; the percentages of sodium phosphate refer to anhydrous chemical. The crystal water of sodium phosphate was accounted for in calculating the amount of mix water required.

### Molding moisture contents

The moisture density, and moisture, 7 day cured plus 1 day immersed strength relationships of the three soils treated with a 2% and 12% of each lime plus 0%, 0.5% and 2% of sodium carbonate, and with 4%, 6% and 8% of each lime plus 0%, 0.5% and 2% of sodium hydroxide, were determined[1]. The moisture density and moisture immersed strength curves showed that for each mixture the optimum molding moisture contents for maximum dry density and for maximum immersed strength were nearly

the same. Hence it was decided to mold all test specimens at optimum moisture for maximum density.

**Molding of specimens**

Each mixture was molded into cylindrical shaped specimens, 2 ± 0.05 inches high and 2 inches in diameter having a density near 100% standard Proctor. The drop-hammer compaction apparatus and the molding method used have been described[7].

**Curing of specimens**

Specimens made for evaluating the effect of chemical additives on the strength of soil and lime mixtures were cured for 7 and 28 days in a moist curing room maintained at a relative humidity of 90% or higher. The temperature in the moist curing room was within a few degrees of 70°F, except for the mixes with sodium hydroxide, which were cured at a temperature between 75 and 80°F, due to malfunctioning of the temperature control system. After being molded and prior to storing in the curing room, each specimen was sealed in wax paper to reduce the loss of moisture and to prevent carbon dioxide of the air from reacting with the lime in the specimens during curing.

The specimens for the variable temperature curing studies, after being molded, were sealed in wax paper. Those treated with sodium hydroxide were further wrapped with Saran wrap and aluminum foil. They were then placed in metal containers having tight fitting lids. A small can of water inside each container maintained the relative humidity near 100 percent. Little loss in weight by the specimens was observed, indicating little or no loss of moisture during curing. The specimens for curing temperature versus immersed strength tests were cured for 3, 7, 14 and 28 days at temperatures of 50, 70, 100 and 140°F. Additional sets of specimens were cured 7 days at each temperature in preparation for freeze thaw testing.

**Immersed strength testing**

The cured specimens were immersed in distilled water for 24 hours and then tested for unconfined compressive strength. The rate of loading specimens in the testing machine was 0.1 inches per minute. The maximum load causing failure was taken as the immersed strength of the specimen. Strength values reported in psi are the average of three test specimens; the difference between the individual values and the average value rarely exceeded 10% of the average value.

**Durability testing**

A modified British standard freeze thaw test (B.S. 1924-1957)[1], was used to evaluate the durability of selected mixtures. Two identical 2 by 2

inch specimens from each mixture were cured 7 days at each of the previously mentioned temperatures, then immersed in distilled water at room temperature for 24 hours. One specimen, designated the control specimen, was left immersed for 14 more days; and the other specimen, designated the freeze-thaw specimen, was exposed alternately to temperatures of 23 ± 2° F (16 hrs.) and 77 ± 4° F (8 hrs.) for 14 cycles, each cycle lasting 24 hours. (A vacuum flask specimen container was used to cause freezing to occur from the top down and to supply unfrozen water to the bottom of the specimen throughout the test.) After these treatments, the unconfined compressive strength of the freeze-thaw specimen ($p_f$) and of the control specimen ($p_c$) were determined. These values were used to evaluate the durability of the stabilized soils. The index of the resistance to the effect of freezing ($R_f$) was calculated from the formula:

$$R_f = \frac{100\ p_f}{p_c} \quad (\%)$$

## TEST RESULTS AND INTERPRETATIONS

### Effect of lime on immersed strength

*Dolomitic hydrated lime.* Specimens of Kansan till, plastic loess, and Kansan gumbotil treated with 2% to 12 or 14% dolomitic lime, in 2% increments, were tested for immersed strength (unconfined compressive strength after 24 hours immersion) after 7 and 28 days moist curing at approximately 70°F and 90+% R.H. (figure 1). The general trend of the data is similar for each soil at each curing period, showing low immersed strengths at low lime contents, near maximum strengths at intermediate lime contents, and little or no further increase of strength at higher lime contents. Recommended base course mix designs for roads might be: Kansan till plus 4% lime, plastic loess plus 6% lime, Kansan gumbotil plus 8% lime, but there might be an advantage in using more lime with the latter soil. These mixtures gave 7 and 28 day cured immersed strengths of 163 and 276 psi (Kansan till), 77 and 228 psi (plastic loess), 141 and 203 psi (Kansan gumbotil). The use of higher lime contents with Kansan till and Kansan gumbotil gave 28 day strengths of about 300 psi.

*Calcitic hydrated lime.* The results of comparable tests using calcitic lime instead of dolomitic lime show that immersed strengths reached a fairly well-defined maximum value at fairly low lime contents and showed no increase at the higher lime contents used (figure 2). Mix designs probably would be: Kansan till plus 4% lime, plastic loess plus 4% lime, Kansan gumbotil plus 6% lime. Corresponding 7 and 28 day cured immersed strengths were: 133 and 209 psi (Kansan till), 54 and 153 psi (plastic loess), 69 and 151 psi (Kansan gumbotil).

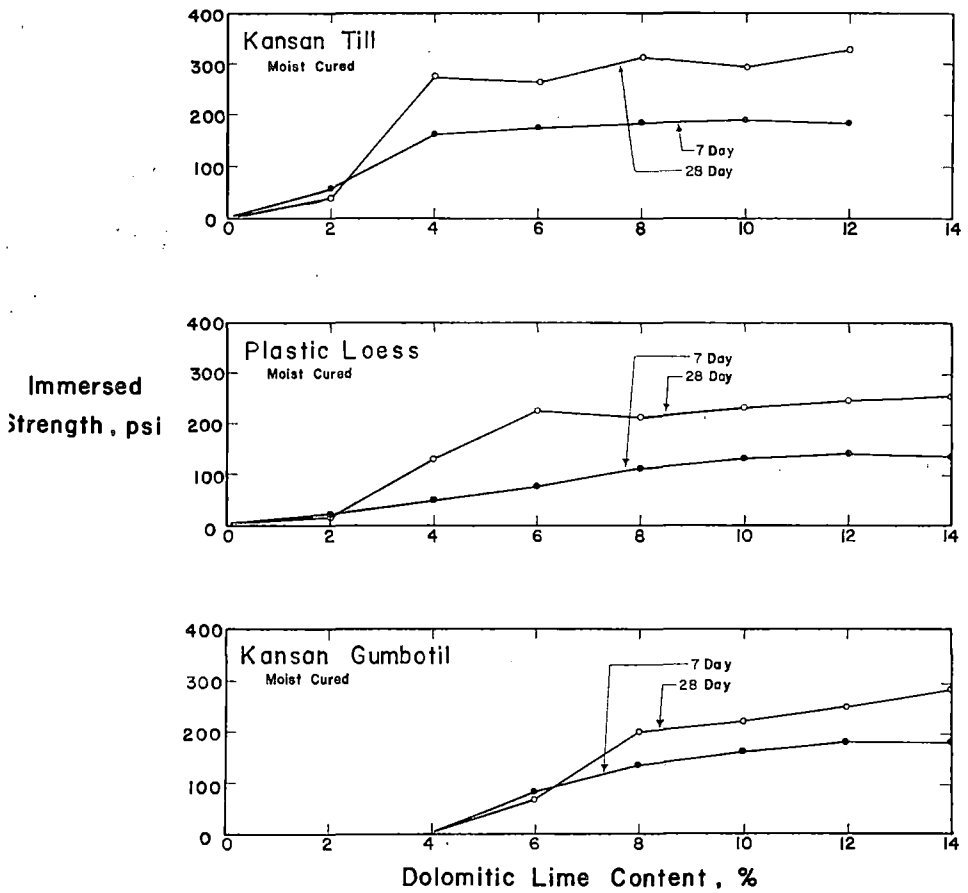


**Fig. 1. Immersed strength-dolomitic lime content relationships for Kansan till, plastic loess and Kansan gumbotil.**

*Summary—Dolomitic lime vs. calcitic lime.* The immersed strengths obtainable with treatments of dolomitic monohydrate (Type N) lime indicate that the three clay soils could probably be satisfactorily stabilized for base courses of roads in Iowa, providing construction was completed early enough to take advantage of high summer temperatures. Mixtures of the soils with calcitic lime probably would not be satisfactory for base courses, but should be satisfactory for subbases or subgrade treatments.

**Effect of lime plus chemical additives on immersed strength**

*Dolomitic hydrated lime plus sodium carbonate or sodium phosphate.* Specimens of Kansan till treated with 4, 6 and 8% of dolomitic lime plus 0, 0.25, 0.5, 1, 2 and 4% of each chemical were cured 7 and 28 days at approximately 70° F and 90+ R.H., immersed in distilled water for 24 hours, then tested for unconfined compressive strength. Test results indicated that the use

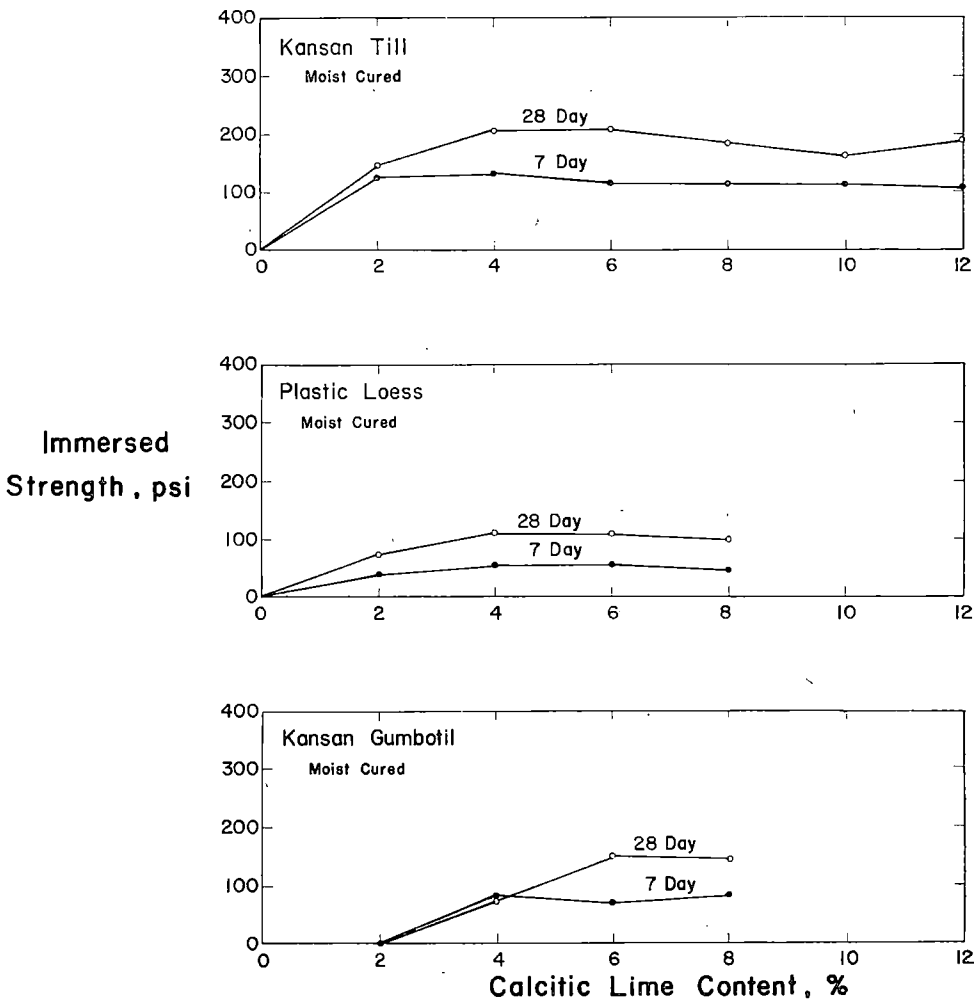


**Fig. 2. Immersed strength-calcitic lime content relationships for Kansan till, plastic loess and Kansan gumbotil.**

of sodium carbonate or sodium phosphate in Kansas till, dolomitic lime mixtures caused a marked decrease in immersed strength. Therefore these chemicals were not evaluated with the other two soils.

*Calcitic hydrated lime plus sodium carbonate or sodium phosphate.* Specimens of Kansan till with combination treatments of calcitic lime and each chemical, proportioned the same as the combination dolomitic lime, chemical treatments, gave fair immersed strength results (figure 3). Definite optimum combination treatments are indicated, the best being 6% lime plus 2% sodium carbonate, and 6% lime plus 2% sodium phosphate. The former

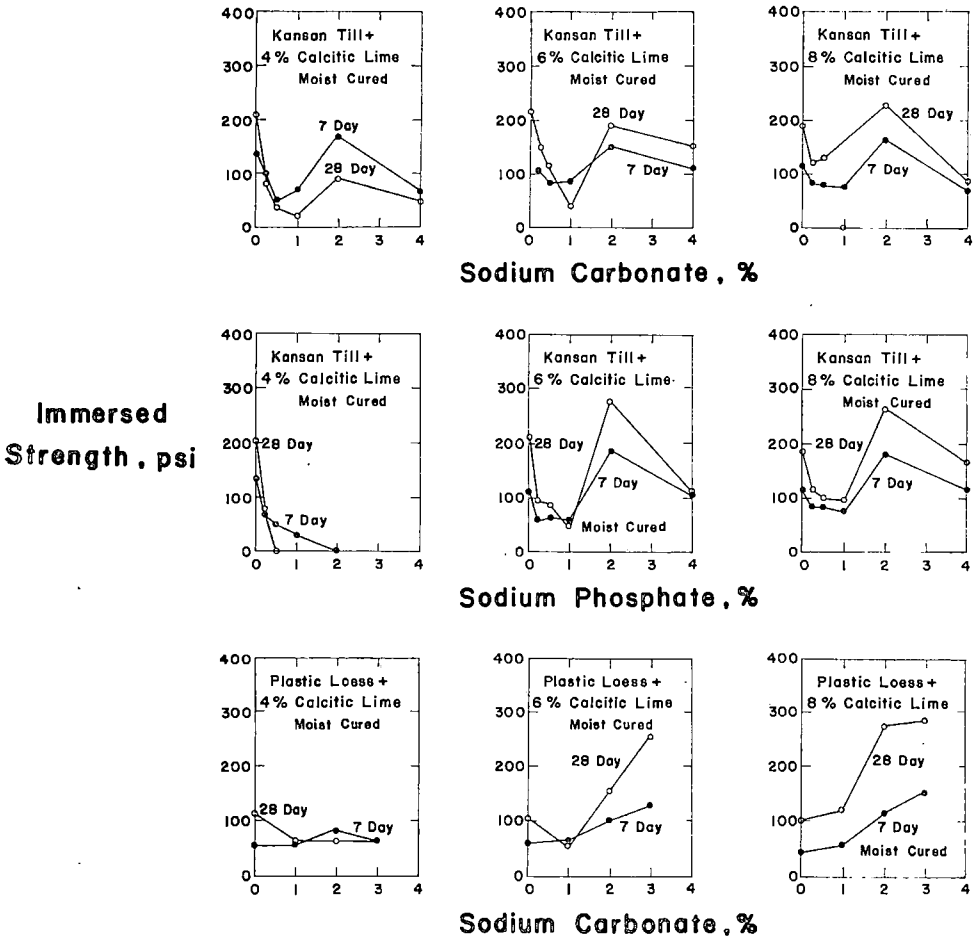


**Fig. 3. Immersed strength-sodium carbonate or sodium phosphate content relationships for calcitic lime-treated Kansan till and plastic loess.**

treatment gave 7 and 28 day cured immersed strengths of 251 and 292 psi; the latter treatment gave 183 and 278 psi, compared with 117 and 210 psi with 6% lime and 0% chemical. Since sodium phosphate is more expensive than sodium carbonate and did not produce as high strengths, it was not evaluated further.

The effects of additions of 1, 2 and 3% sodium carbonate on the immersed strength of plastic loess, lime mixtures were determined (figure 3). Smaller amounts of sodium carbonate were not tried because of the adverse effects observed with Kansan till, lime mixtures. A good combination treatment of plastic loess was 6% lime plus 3% sodium carbonate; the 7 and 28 day cured immersed strengths were 130 and 256 psi. Another, slightly

better but more costly, was 8% lime plus 3% sodium carbonate; the 7 and 28 day strengths were 143 and 291 psi. For comparison, the 7 and 28 day strengths of plastic loess, 6% lime were 56 and 109 psi, and of plastic loess, 8% lime were 45 and 99 psi.

The combination treatments used with plastic loess also were tried with Kansan gumbotil. Test results are not shown, since all amounts of sodium carbonate decreased the immersed strength of Kansan gumbotil, lime mixtures, both after 7 and 28 day curing.

*Summary—sodium carbonate and sodium phosphate.* The foregoing test results indicated that the search for a chemical additive to improve clayey soil, lime mixtures was not ended. Additives of sodium carbonate and sodium phosphate decreased the immersed strengths of the soil, dolomitic lime mixtures. Optimum combination treatments of lime and chemical gave marked improvements to the immersed strengths of Kansan till and plastic loess, but use of a combination treatment other than the optimum generally had an adverse effect on strength. Sodium carbonate was judged more promising than sodium phosphate, but even sodium carbonate decreased the immersed strength of Kansan gumbotil-calcitic lime mixtures. Thus it was decided to continue the search for a more versatile and beneficial additive. Exploratory tests indicated that sodium hydroxide was a likely prospect.

*Each lime plus sodium hydroxide.* The sodium hydroxide treatments of each soil, lime mixture were 0, 0.5, 1, 2 and 3 or 4%. The following mixtures were so treated: each soil plus 4, 6 and 8% dolomitic lime, and each soil plus 4, 6 and 8% calcitic lime. Specimens were moist cured 7 and 28 days at temperatures in the range 75° to 80°F, which were 5° to 10°F higher than the curing temperature of the specimens in the previously discussed tests. The resulting improvements of immersed strength are seen by comparing the strength values for soil, lime mixtures (0% sodium hydroxide) (figures 4, 5, 6) with those for identical mixtures (figure 3).

*Kansan till.* Test results with Kansan till showed that sodium hydroxide, in amounts of 1 to 4%, improved the 7 and 28 day cured immersed strengths of both dolomitic lime and calcitic lime treated soil mixtures; 0.5% sodium hydroxide was detrimental to the strengths of all mixtures (figure 4). There was generally an optimum amount of chemical for each Kansan till-lime mixture, and strength improvements at and near the optimum treatment were exceptional. For example, the 7 day strength of the Kansan till-6% calcitic lime mixture was 135 psi; 1% sodium hydroxide boosted it to 363 psi. The 28 day strength was improved even more: 240 psi without chemical, 582 psi with 1% chemical. Sodium hydroxide treatments made possible 7 day strengths near or above 350 psi with all Kansan till-lime mixtures regardless of the type of lime; the same treatments produced 28 day strengths of 500 psi or more. A comparison of the following mix designs, which might be considered satisfactory for a road base course, illustrates

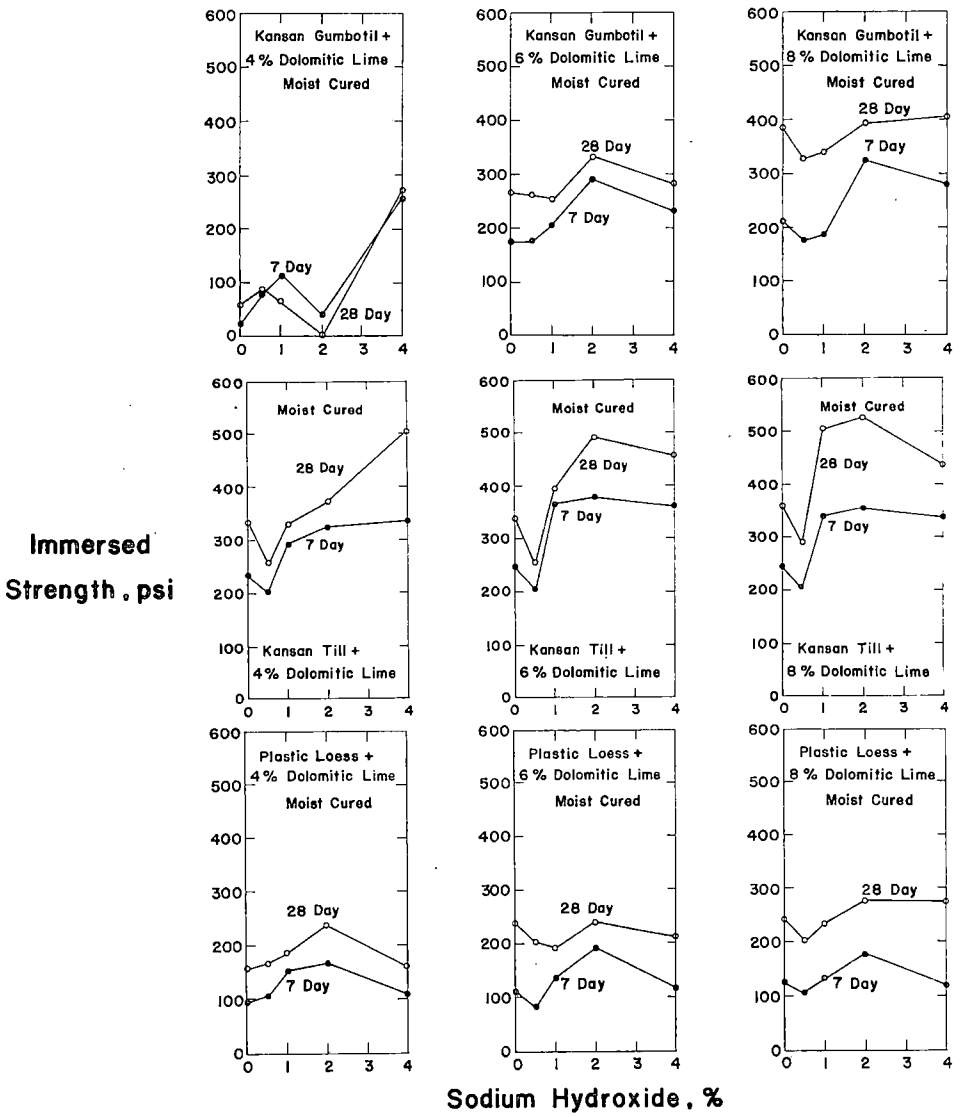


**Fig. 4. Immersed strength-sodium hydroxide content relationships for lime-treated dolomitic Kansan till.**

the possibilities for economical stabilization of Kansan till: 6% dolomitic lime (marginal), 6% dolomitic lime + 2% NaOH (safe), 4% calcitic lime + 1% NaOH (safe).

*Plastic loess.* Test results with plastic loess (figure 5) indicate that although the optimum amounts of sodium hydroxide were generally beneficial to immersed strength of dolomitic lime treated mixtures, best

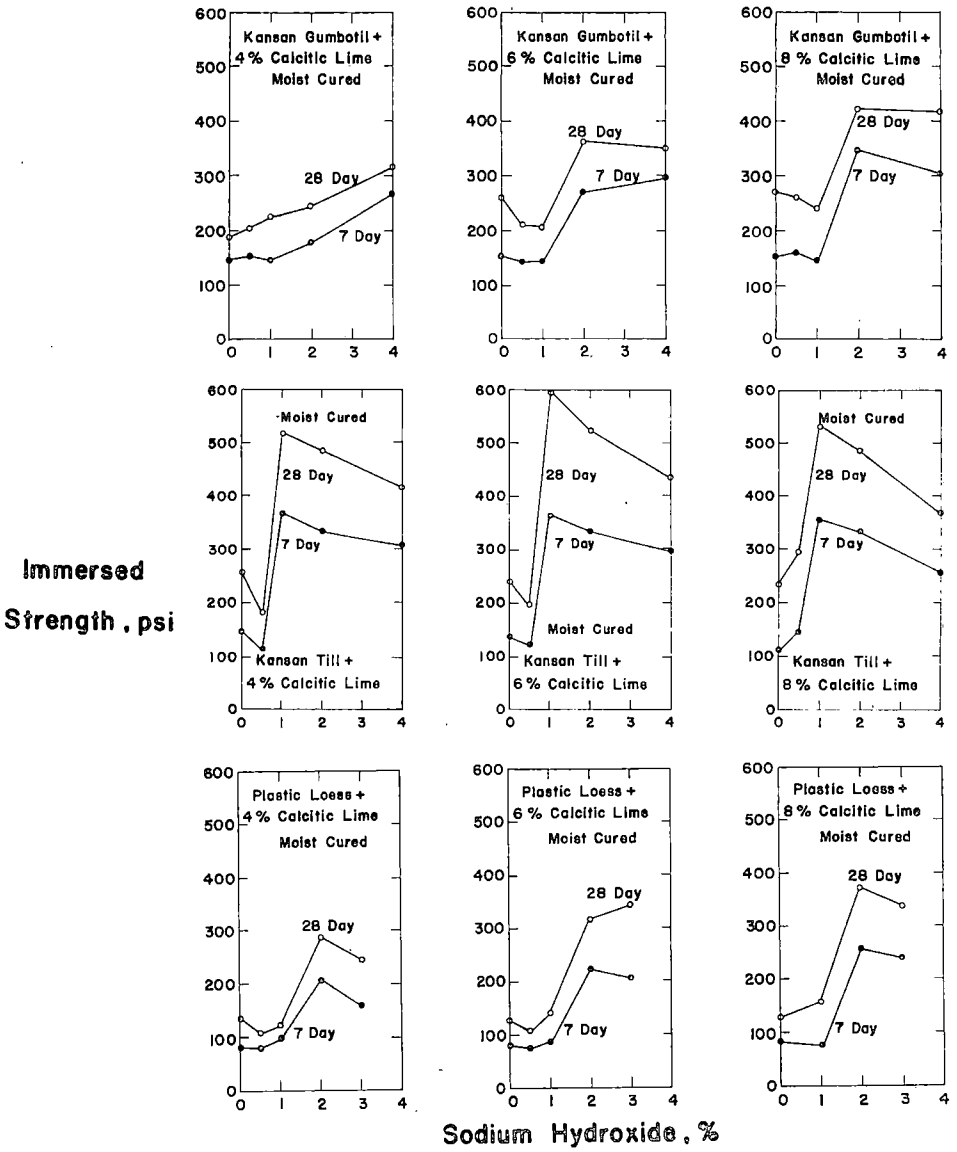


Fig. 5. Immersed strength-sodium hydroxide content relationships for calcitic lime-treated plastic loess.

strength improvements were obtained with the calcitic lime treated mixtures, which needed the most improvement. It is evident that the extent of improvement of plastic loess and lime mixtures was not as exceptional as with Kansan till and lime mixtures, but economical mix designs which may meet base course requirements are made possible by use of sodium

hydroxide. For example, plastic loess plus 6 or 8% calcitic lime and 2% NaOH look promising. Plastic loess plus 8% or 10% dolomitic lime and 2% NaOH would probably give equivalent performance, although no data is shown for 10% dolomitic lime.

*Kansan gumbotil.* The optimum amount of sodium hydroxide for each Kansan gumbotil-lime mixture greatly improved 7 day cured immersed strength, and, in the case of the calcitic lime mixtures, also 28 day strength. The 28 day strengths of the dolomitic lime mixtures did not show such marked improvements. For example, 2% sodium hydroxide increased the 7 and 28 day strengths of the 8% calcitic lime treated mixture from 155 and 271 psi to 348 and 421 psi, respectively; and the same amount of chemical increased the 7 and 28 day strengths of the 8% dolomitic lime treated mixture from 211 and 382 psi to 324 and 390 psi, respectively. Satisfactory mix designs for road base courses might be with: 8% dolomitic lime, 6 or 8% dolomitic lime plus 2% NaOH, 6 or 8% calcitic lime plus 2% NaOH.

*Summary—sodium hydroxide.* Sodium hydroxide may be the much needed chemical activator for pozzolanic reactions in clayey soil and lime mixtures. The clayey soils used in the tests contained montmorillonite as the dominant clay mineral (table II), and hence represent about as difficult materials to stabilize as will be found anywhere. Work now in progress will show whether combination treatments of lime and sodium hydroxide are equally beneficial to soils dominated by other clay materials.

The test data indicate that most montmorillonitic clay soils can probably be stabilized satisfactorily with lime and sodium hydroxide to meet minimum road base course strength requirements. Apparently the amounts of lime and chemical needed vary only slightly with the amount of clay in the soil, since the clay (0.005 mm) contents of the soils used in the tests covered a wide range (38.5 to 70.5%), and best immersed strengths generally were obtained with 6 or 8% lime or 1 or 2% NaOH. Lime contents higher than 8% were not tried, but they may give added strength to soils such as plastic loess and Kansan gumbotil. The exceptional response of the Kansan till to lime NaOH treatments may be related to its gradation: approximately equal parts by weight of sand, silt and clay sizes. Plastic loess, with approximately the same amount of clay as Kansan till, but no sand, was improved to a considerably lesser extent by the same treatments.

A most encouraging finding was that sodium hydroxide made the calcitic hydrated lime equally or more reactive than the dolomitic monohydrate (type N) lime with the clayey soils. It is hoped that further study will confirm that sodium hydroxide is an equalizer for hydrated limes in soil stabilization, thus eliminating the need for specifying dolomitic monohydrate (type N) lime for base course stabilization in Iowa.

*Effect of curing temperature on immersed strength.* The following four treatments of Kansan till were selected for the immersed strength curing temperature relationship study: 6% dolomitic lime, 6% calcitic lime, 6%

calcitic lime plus 2.25% sodium carbonate, 6% calcitic lime plus 1% sodium hydroxide. Specimens of these mixtures were cured at 50°, 70°, 100° and 140° F for 3, 7, 14 and 28 days. Cured specimens were immersed for 24 hours, then tested for unconfined compressive strength. The test results are shown as immersed strength versus curing time for each temperature, and as immersed strength versus curing temperature for each time (figure 6).

*Kansan till plus 6% dolomitic lime.* At each curing temperature, immersed strength increased with curing time up to 14 days; thereafter except at 50° F, the rate of increase was less. At 140° F, 28 day strength was less

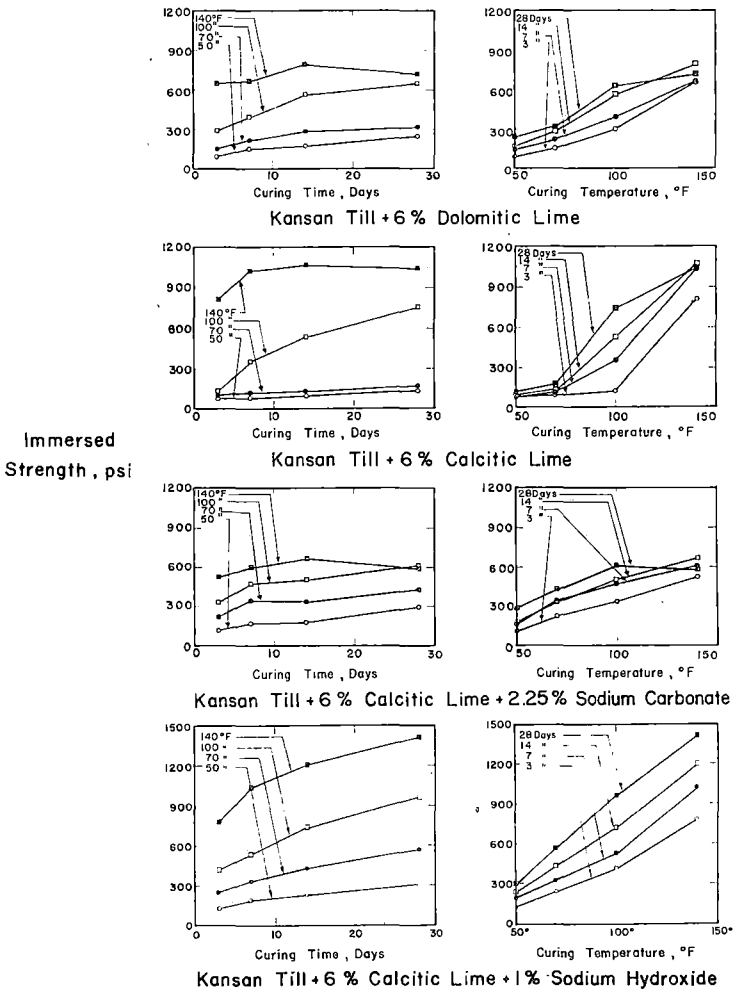


**Fig. 6. Immersed strength-curing temperature and curing time relationships for Kansan till treated with 6% dolomitic lime, 6% calcitic lime, 6% calcitic lime plus 2.25% sodium carbonate, or 6% calcitic lime plus 1% sodium hydroxide.**

than 14 day strength. The greatest rate of strength gain up to 28 days was at 100° F; the immersed strength increased from 300 psi at 2 days to over 600 psi at 28 days. The least change of strength was at 140° F; at this temperature the soil lime reaction may have been nearly completed at 2 days.

It appears that for emergency mix design purposes, accelerated curing for 3 or 7 days at 140° F could be used to obtain a fairly accurate estimate of immersed strength after 28 days curing at 100° F. Similarly, 3 days curing at 100° F was comparable with 28 days curing at 70° F.

The plots of immersed strength versus curing temperature further illustrate the beneficial effect of high temperature curing on strength after each curing period. At 7 days, where the relationship was nearly linear, the increase in immersed strength per degree Fahrenheit increase in curing temperature was roughly 6 psi. The effect of curing temperature on 14 and 28 day strengths, if anything, was even greater.

There would be little question of the adequacy of this mixture for a road base in regions having atmospheric temperatures of 100° F during the construction season; three days of such curing would probably suffice.

*Kansan till plus 6% calcitic lime.* The results of tests with this mixture may explain why calcitic lime is considered satisfactory for soil lime stabilization in southern climates, and why it may not be equally satisfactory for base course stabilization in the north. At curing temperature of 50 and 70° F, immersed strengths up to 28 days are too low for base courses, if the curing period is to be followed by freeze-thaw cycles. At 100° F the hardening mechanism was tremendously accelerated, and immersed strength increased from about 140 psi at 2 days to about 750 psi at 28 days, very adequate for base courses in any climate. At 140° F the hardening was about completed at 7 days, and the immersed strength was over 1000 psi.

Indications are that for speedy mix design the immersed strength after 3 days curing at 140° F could be used to predict immersed strength after 28 days curing at 100° F. Also, 3 day strength at 100° F seems indicative of 28 day strength at 70° F.

The immersed strength versus curing temperature curves also illustrate the great benefit to strength gain of curing temperatures above 70° F. Fourteen day strength showed a linear relationship in the temperature range between 70° and 140° F; each degree Fahrenheit increase in temperature increased (14 day immersed) strength about 13 psi.

*Kansan till plus 6% calcitic lime and 2.25% sodium carbonate.* A comparison of the plotted test data for this mixture with that for the mixture of Kansan till plus 6% calcitic lime (0% sodium carbonate) indicates that use of the chemical additive would be worthwhile only when early strength up to 3 days is of paramount importance, or when curing would be at temperatures around 50° F to 70° F. At 100° F curing there was little or no benefit to immersed strength, except at 3 days, from use of the chemical, and at 140° F the chemical additive was definitely detrimental to strength at all

ages. It appears that either high temperatures or sodium carbonate can accelerate the pozzolanic reactions. Of the two, high temperature is more effective.

*Kansan till plus 6% calcitic lime and 1% sodium hydroxide.* This mixture showed exceptionally good immersed strength gains with increase of curing from 137 psi at 3 days to 297 psi at 28 days; at 70° F, this increase was from 250 psi to 567 psi; at 100° F, this increase was from 418 psi to 960 psi; at 140° F, this increase was from 780 psi to 1426 psi. It is of interest to note that 7 day strength at 140° F is approximately equal to 28 day strength at 100° F; and that 7 day strength at 100° F is approximately equal to 28 day strength at 70° F.

The immersed strength versus curing temperature relationship was nearly linear for each curing period. For 7 and 28 day curing, each degree Fahrenheit increase of temperature increased immersed strength about 10 and 13 psi, respectively.

Comparison of the strength data for Kansan till plus 6% calcitic lime with and without sodium hydroxide treatment, indicates that since both heat and sodium hydroxide benefit the hardening mechanism, the sodium hydroxide treatment would be advantageous for base course construction in any climate, but particularly so in warm climates, where appreciable reductions in lime content might be made possible.

*Summary—curing temperature.* High temperature curing greatly accelerated the hardening mechanism of Kansan till stabilized with 6% of either dolomitic monohydrate (Type N) lime or calcitic hydrated lime. At 100° F, both mixtures gave immersed strengths which exceeded 300 and 600 psi after 7 day and 28 day curing, respectively.

The 2.25% additive of sodium carbonate to the Kansan till calcitic lime mixture accelerated hardening at curing temperatures of 50° and 70° F; but at 100° F curing the chemical was beneficial only to 3 and 7 day strengths; and at 140° F curing, the chemical greatly nullified the beneficial effect of heat on strength at all periods up to 28 days. The adverse effects of sodium carbonate on strength may have been due to heat-induced swelling, and consequent weakening by disruptive internal pressures.

The addition of 1% sodium hydroxide to the Kansan till and 6% calcitic lime mixture acted as an excellent accelerator for the hardening mechanism, both at normal and high curing temperatures. Thus its beneficial effects were in addition to those due to heat, a desirable trait in a chemical accelerator.

**Durability of lime, or lime and chemical treated Kansan till**

The four mixtures used to study immersed strength curing temperature relationships were further evaluated by the modified British freeze-thaw test[1]. The control and freeze-thaw specimens of each mixture were moist cured 7 days at 50°, 70°, 100° or 140° F prior to evaluation. Cured

TABLE IV. RESULTS OF FREEZE-THAW TESTS BY MODIFIED BRITISH METHOD, B.S. 1924: 1957

| Additive(s) | As-molded Density pcf | Curing temperature (7 days), deg. F. | $p_f$ psi | $p_c$ psi | $R_f$,* % |
|---|---|---|---|---|---|
| 6% dolomitic monohydrate (Type N) lime | 107.5 | 50 | 177 | 277 | 65 |
| | " | 70 | 235 | 254 | 90 |
| | " | 100 | 367 | 392 | 95 |
| | " | 140 | 617 | 653 | 95 |
| 6% calcitic hydrated lime | 106.2 | 50 | 109 | 138 | 80 |
| | " | 70 | 103 | 122 | 85 |
| | " | 100 | 277 | 289 | 95 |
| | " | 140 | 798 | 974 | 80 |
| 6% calcitic hydrated lime plus 2.25% sodium carbonate | 107.0 | 50 | 225 | 351 | 65 |
| | " | 70 | 322 | 418 | 75 |
| | " | 100 | 421 | 472 | 90 |
| | " | 140 | 650 | 597 | 110 |
| 6% calcitic hydrated lime plus 1% sodium hydroxide | 110.2 | 50 | 212 | 357 | 60 |
| | " | 70 | 341 | 420 | 80 |
| | " | 100 | 449 | 599 | 75 |
| | " | 140 | 981 | 828 | 120 |

*$R_f = \frac{100 p_f}{p_c}$ reported to nearest 5%.

control specimens were immersed in distilled water for 15 days, then tested for unconfined compressive strength ($p_c$). Cured freeze-thaw specimens were immersed for 1 day, subjected to 14 freeze-thaw cycles (14 days), then tested for unconfined compressive strength ($p_f$). Test results and the calculated indices of the resistance to the effect of freezing ($R_f$) are given in table IV.

*Criteria of durability.* The criteria which may be used to evaluate the results of the modified British freeze-thaw test are: (1) the $R_f$ value should equal or exceed a specified value, (2) the individual $p_f$ and $p_c$ values should equal or exceed a specified value. The following minimum values appear to be conservative for the design of stabilized mixtures for road base courses in climates such as that in Iowa: $R_f = 80\%$, $p_c = 250$ psi, $p_f = 250$ psi. A discussion of these criteria may be found in reference[1]. When both $p_c$ and $p_f$ values greatly exceed 250 psi, a lower minimum $R_f$ may be permissible, for example, 75% as used in England.

*Interpretation of test results.* High temperature curing improved the freeze-thaw resistance of specimens of the Kansan till and 6% dolomitic lime mixture, and those cured at 100° and 140° F showed satisfactory durability. The specimens cured at 70° F were indicative of marginal quality for road bases, and those cured at 50° F failed to meet the recommended minimum durability values. Thus a road base of this mixture should have some hot weather curing before undergoing wintering in Iowa, and completion of construction in August, at the latest, would be recommended.

If the mixture of Kansan till and 6% calcitic lime is judged on the basis of test results after 50° or 70° F curing, it does not meet base course requirements. However specimens cured at 100° and 140° F proved more than adequate, indicating that this mixture might make a satisfactory road base in a warm climate.

For northern climates where temperatures of 70° F or higher prevail during the construction season, the test results in table IV indicate that the Kansan till and 6% calcitic lime mixture could be brought up to base course quality by the addition of either 2.25% sodium carbonate or 1% sodium hydroxide. Of the two chemical accelerators, sodium hydroxide would permit the greatest flexibility in construction planning and the longest construction season.

*Summary—durability.* The durability test data for Kansan till indicate that, whereas dolomitic monohydrate (type N) lime stabilization may give adequate durability for road bases in Iowa, calcitic hydrated lime stabilization may not, unless a chemical accelerator is used. Sodium hydroxide appears especially promising for this purpose.

## CONCLUSIONS

1. Addition of a small amount of sodium hydroxide to mixtures of montmorillonitic clay soil and lime acts as an effective accelerator for the hardening mechanism of the compacted mixture. The optimum sodium hydroxide treatment is about 1 to 2%, based upon the dry weight of the soil component.
2. Sodium carbonate and sodium phosphate are not as promising as sodium hydroxide for improving lime stabilization of montmorillonitic clay soils, particularly when the lime is dolomitic monohydrate (Type N).
3. The magnitude of strength improvement up to 28 days, due to the use of sodium hydroxide, varies with the composition of the lime and the clay soil; calcitic (high calcium) hydrated lime may give somewhat better results than dolomitic monohydrate (Type N) lime, but the presence of sand-size quartz in the soil is definitely beneficial.
4. Heat also accelerates the hardening of compacted mixtures of montmorillonitic clay soil and lime, and temperatures around 100° F or higher during the early curing period are extremely beneficial to strength and durability, the beneficial effects apparently supplementing those obtainable from the use of sodium hydroxide as an additive.
5. Dolomitic monohydrate (Type N) lime should be used for lime stabilization of montmorillonitic clay soils for road base courses in Iowa or regions of similar or more severe climate, unless an additive of sodium hydroxide is specified; in which case, either the dolomitic lime or a calcitic hydrated lime may be used. In Iowa, without sodium hydroxide additive, calcitic lime appears satisfactory only for subbase or subgrade stabilization.

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# ISOLATION AND INVESTIGATION OF A LIME MONTMORILLONITE CRYSTALLINE REACTION PRODUCT

by

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(Highway Research Board Proceedings. 1961.)

## INTRODUCTION

During previous research it was noted that mixes of lime and a soil rich in montmorillonite clay mixed with water above the liquid limit would within several days apparently be dried below the liquid limit with no loss in weight. To determine what was occurring, a portion of this sample was subjected to X-ray diffractometer analysis. Strong indications of a new reaction product were observed, but the product could not be found under the microscope.

### Objective

The object of this paper is to investigate one phase of the mechanisms producing cementation by attempting to isolate and determine the properties of a crystalline reaction product associated with the action of lime with clay.

From the results obtained in this research, other studies have developed to explore additional crystalline products and are now being pursued at the Iowa Engineering Experiment Station Soil Research Laboratory. When this research is completed and the information is evaluated, it is expected that the relationships involved in the formation of new products and the breakdown of the original clay lattice can be determined.

### Procedures

The research undertaken was first to investigate various lime, soil, and water systems varying each of the three parameters one at a time to determine the best phase relationships for growing euhedral crystals of sufficient size to be observed under the microscope, second to isolate these crystals, and third to determine their physical and chemical properties.

In the systems investigations a large number of mixtures were prepared and allowed to cure for periods from two days to eight months before

examination. Isolating the crystals was done under the microscope, and the determination of their properties was undertaken primarily by microscopic and X-ray methods.

## MATERIALS AND EQUIPMENT

### Soils

The choice of the five soils used in this investigation was based on the type, purity, and amount of the principal clay mineral present and the availability of the soil. Each of the soils is identified by a letter designating the principal clay mineral and by a number indicating the percentage content of the soil less than two microns in diameter. For example, the designation, M-75, indicates a soil containing montmorillonite as the principal clay mineral with 75% of this soil less than 2 microns in effective diameter (tables I, II).

### Clays

Two different clays were used, one a bentonite and the other a kaolinite. These particular clays were selected because they contained only small amounts of impurities.

### Lime

Reagent grade calcitic hydrated lime, $Ca(OH)_2$, was used to minimize compositional variables. Individual one pound bottles of lime were kept

TABLE I. SOIL SITE CHARACTERISTICS

| Sample | Location | Classification | Soil series and horizon | Sampling depth, in. |
|---|---|---|---|---|
| M-67 | Keokuk County, Iowa | Post Kansan paleosol | Mahaska, fossil B horizon | 91 - 101 |
| M-51 | Harris County, Texas | Coastal plane deposit, largely deltaic | Lake Charles, probably B horizon | 39 - 144 |
| I-44 | Monroe County, Michigan | Probably Wisconsin-age glacial till | Unknown series, probably C horizon | Unknown |
| I-41 | Livingston County, Ill. | Wisconsin-age glacial till | Clarence, C horizon | 46 - 56 |
| K-30 | Durham County, North Carolina | Residual soil over medium grained biotite granite | Durham, B horizon | 24" below bottom of A horizon |
| Bentonite | Otay, California | Pliocene age transported altered ash | San Diego formation | 120 - 216 |
| Kaolinite | Bath, South Carolina | Cretaceous age | Upper Hamburg formation | 40 - 100 ft. |

TABLE II. PROPERTIES OF SOILS AND CLAY

| Sample | M-67 | M-51 | IC-44 | IC-41 | K-30 | Bentonite |
|---|---|---|---|---|---|---|
| I.E.E.S. designation | 528-8 | AR-3 | AR-4 | AR-8 | AR-6 | ..... |
| Textural composition,* 2% | | | | | | |
| Gravel (> 72 mm) | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | |
| Sand (2 - 0.074 mm) | 16.6 | 3.0 | 7.0 | 10.0 | 45.2 | |
| Silt (74 - 5$\mu$) | 15.5 | 36.0 | 36.0 | 38.0 | 18.3 | |
| Clay (< 5$\mu$) | 70.5 | 61.0 | 57.0 | 52.0 | 36.5 | |
| Clay (< 2$\mu$) | 67.0 | 51.0 | 44.0 | 41.0 | 30.0 | |
| Physical properties | | | | | | |
| Liquid limit, % | 76.6 | 64.6 | 44.0 | 35.5 | 51.0 | 87.0 |
| Plastic limit, % | 25.6 | 17.6 | 21.1 | 17.5 | 25.5 | 52.2 |
| Plasticity index | 50.0 | 47.0 | 22.9 | 18.0 | 25.5 | 34.8 |
| Chemical properties | | | | | | |
| pH | 7.1 | 8.2 | 8.4 | | 5.7 | |
| C.E.C. (soil passing No. 10 sieve, m.e./100 gm) | | 27.5 | 14.5 | | 8.4 | |
| C.E.C. (soil passing No. 40 sieve, m.e./100 gm) | 41.0 | 33.1 | 13.4 | | 13.5 | |
| Carbonates, % | .8 | 16.1 | 7.2 | | 0.1 | |
| Organic matter, % | .2 | 0.1 | 0.6 | | 0.1 | |
| Predominant clay mineral† | M | M | I&C | I&C | K | M |
| Classification | | | | | | |
| Textural‡ | Clay | Clay | Clay | Clay | Clay | Clay |
| Unified | CH | CH | CL | CL | CH-CL | CH |
| BPR (AASHO) | A-7-6(20) | A-7-6(20) | A-6-6(14) | A-6-(11) | A-7-6(11) | A-7-6(20) |

*Textural gradation tests were performed only on the soil fraction passing the No. 10 sieve. All soils used contained less than 5% gravel.

†Symbols are M—montmorillonite, I—illite, C—chlorite, and K—kaolinite. Determinations were made by X-ray diffraction analysis.

‡U.S.D.A. textural classification was used.

sealed until immediately before use to prevent carbonation of the lime by the carbon dioxide in the air.

### Fly Ash

Fly ash is "the finely divided residue that results from the combustion of ground or powdered coal and is transported from the boiler by flue gasses." The fly ash used in this study was collected by a Cottrell precipitator at the St. Clair power plant of the Detroit Edison Co. in Detroit, Michigan.

### Optical Equipment

*Binocular Microscope.* A Bausch and Lomb binocular microscope was used for general investigations of samples. With this instrument a maximum magnification of 90 times is obtained.

A Leitz research model petrographic microscope was used in examining the crystals to determine their index of refraction, interference figure, and optic sign.

### X-Ray Equipment

*Diffractometer.* The General Electric XRD-5 diffractometer was used for general investigations of samples for the presence of crystalline reaction products.

*Debye-Scherrer Camera.* The Debye-Scherrer camera was used to determine lattice constants. The camera, 57.5 mm in diameter and 70 mm in length, is manufactured by Siemens and Halske Aktiengesellschaft, Karlsruhe, Germany. The film measuring device, made by the same company, permits an accuracy of linear measurement of $\pm$ .01 mm.

*Weissenberg Camera.* The Weissenberg apparatus used in this study was manufactured by Otto von der Heyde Co. in Newton Highland, Mass. It was used first to obtain rotation pictures of a single crystal. Following this, Weissenberg photographs were taken to index the reflections and to aid in determining the space group to which the crystal belongs.

## MIXTURE PREPARATIONS AND INVESTIGATIONS

### Preliminary Work

The first indication of the presence of reaction products was found when mixtures of soil M-67, 12% lime, and water (above the liquid limit), which had been prepared for the determination of liquid limits, were subjected to X-ray diffractometer analysis. The results of this analysis showed new X-ray "peaks" occurring at 8.11 and 7.60 Ångstroms. However, in material from 2 by 2 inch specimens compacted at optimum moisture content no such peaks were observable.

Petrographic microscope examinations of the liquid limit mixtures were attempted to isolate the reaction products formed, but no new substances were observed. It then became the object of this phase of the research to attempt to grow euhedral crystals of these reaction products of at least microscopic size and determine their properties. To do this various phase relationships were investigated empirically in trying to produce recognizable crystals.

### Trial Mixtures

*Lime, water, and soil systems*

*Water as the variable parameter.* A reasonable assumption seemed to be that the clay fraction of soil M-67 was the actively participating part of the soil in the reaction with lime. Therefore soil M-67 was shaken through a 325 mesh sieve to obtain the finest fraction of the soil possible through purely mechanical separation. To obtain the most representative sample possible the sieve pan was emptied often, and the material remaining on the 325 sieve was repulverized. This process was repeated until only a negligible amount of soil passed through the sieve during a thirty minute

period. To avoid any process which might change the nature of the clay before being mixed with lime, the material was not washed through the sieve.

This material was then mixed with 20% lime by weight of oven dry soil, and distilled water was added in amounts of 110, 100, 90, 80, 70, 60, 50, 40, 30, and 20% by dry weight of soil plus lime. The materials were mixed in soft plastic containers. After thorough mixing the containers were covered with snap type lids and were placed in a controlled temperature and humidity room (70° F and 95% relative humidity).

After thirty days the containers were removed and a sample of the mixture from each was subjected to X-ray diffractometer analysis (figure 1). Since the mixtures were smoothed into the sample holders by longitudinal strokes with a glass slide, preferred orientation of the particles was attained. From the diffractometer traces it is evident that the reaction products were present in greater quantities at high moisture contents. At moisture contents below 40% the product peaks almost disappeared, which explains why specimens compacted at optimum moisture gave no indication of these reaction products when subjected to X-ray analysis.

One of the two peaks at 8.11 Å is the second order basal reflection from montmorillonite sharpened considerably by the action of absorbed calcium and reaches maximum height at higher moisture contents (figure 1). The 7.59 Å peak is that of a crystalline reaction product.

Examination of the mixtures under the binocular and petrographic microscopes disclosed the formation of euhedral crystals of hexagonal shape. Although these crystals were quite small, the larger ones being of the order of 50$\mu$ across the flats by 5$\mu$ thick, X-ray powder photographs of minute amounts showed them to be the crystals being sought, corresponding to the 7.59 Å peak (figure 1).

Soil M-67 passing the 270 mesh sieve and retained on the 325 mesh sieve, 20% lime, and 100% water were mixed, cured for thirty days, and subjected to X-ray diffractometer analysis. No new peaks were formed, confirming that it was the clay fraction of the soil entering into the reaction.

From the variable water investigation it appeared that mixing samples at high moisture contents would be advantageous, and this was done with the mixtures prepared later.

*Lime as the variable parameter.* Since mixtures should be of high moisture content, the effect of varying the amounts of lime was investigated at a constant moisture content of 105%. Lime was mixed with soil M-67 in quantities of 5, 10, 15, 20, 25, 30, and 50% by weight of dry soil. X-ray diffractometer traces were run on these mixtures after 30 days moist curing. It was found that the height of the new peaks grew successively larger as the percentage of lime was increased to 20%, and above this the height remained relatively constant while the height of the residual calcium hydroxide peak increased. It appeared that the optimum lime content for a

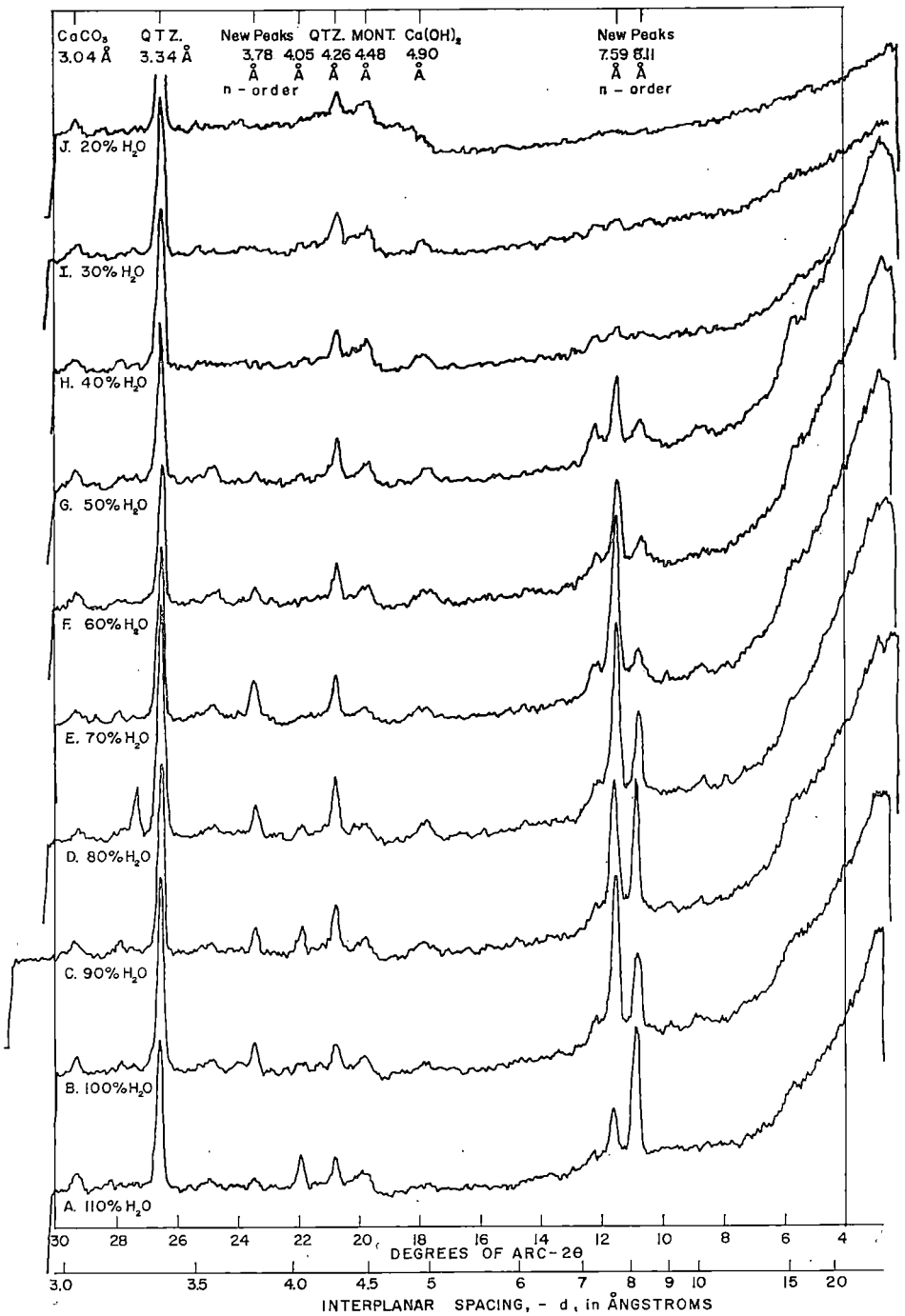


**Fig. 1. Diffractometer charts, mixtures of soil M-67, lime, and variable water after curing for 30 days.**

thirty day period was about 20% for this soil.

Under the microscope, the same extremely small euhedral crystals were observed scattered through the matrix in those samples containing 10% lime or more.

*Soil as the variable parameter.* Other soils were also investigated as to their reaction with lime. These soils included M-51, IC-44, IC-41 and K-30. Of these only the montmorillonite soil M-51 showed any significant height of new peaks. Soil K-30 gave a slight indication and the others none at all. However, these mixtures were allowed to cure for only 30 days. As will be seen later, it would be advantageous to let them cure for six months and then examine them by X-ray diffraction.

**Lime, water, and bentonite investigation**

Since the main component in soil M-67 is montmorillonite clay, and since it is the clay fraction which is reacting with the lime, it seemed logical that better results might be obtained by mixing a bentonite with lime. For this purpose, a bentonite from Otay, California, was selected because of the low amount of impurities it contains. Since the amount of montmorillonite was increased, the amount of lime added was also increased to 40% by dry weight of bentonite. Water was added in the amount of 110% by dry weight of bentonite plus lime. Several samples were made and one was tested after 30 days moist curing. The results of the X-ray analysis on this sample were disappointing in that only a small peak appeared at the expected interplanar spacing, and no crystals could be located with the microscope. The other samples were allowed to remain in the humidity room for seven months before being tested.

When they were examined under the microscope after seven months, crystals which were large in comparison with those obtained previously were found in abundance. The larger ones were about 500$\mu$ across the flats by 20$\mu$ thick. From these the size graded down to submicroscopic (figure 2A).

Since crystal formation was so much more rapid in soil M-67 than in the bentonite, the montmorillonite soil must be either less well crystallized or the soil must contain ions which act as accelerators for the reaction.

**Lime, water, and fly ash investigation**

Since fly ash is well known for its pozzolanic properties, and since it has been used with lime in soil stabilization, a mixture of fly ash plus 30% lime plus 100% water was mixed and placed in the humidity room. After seven months curing the mixture was removed and investigated under the microscope. Large euhedral crystals with geometry similar to those from the bentonite (figure 2B) had been formed throughout the mixture.

The 7.59 Å basal spacing was observed, but a Debye-Scherrer photograph was not made for several months. After several months X-ray analy-

sis showed that the substance had become amorphous, and those crystals produced from bentonite retained their crystallinity.

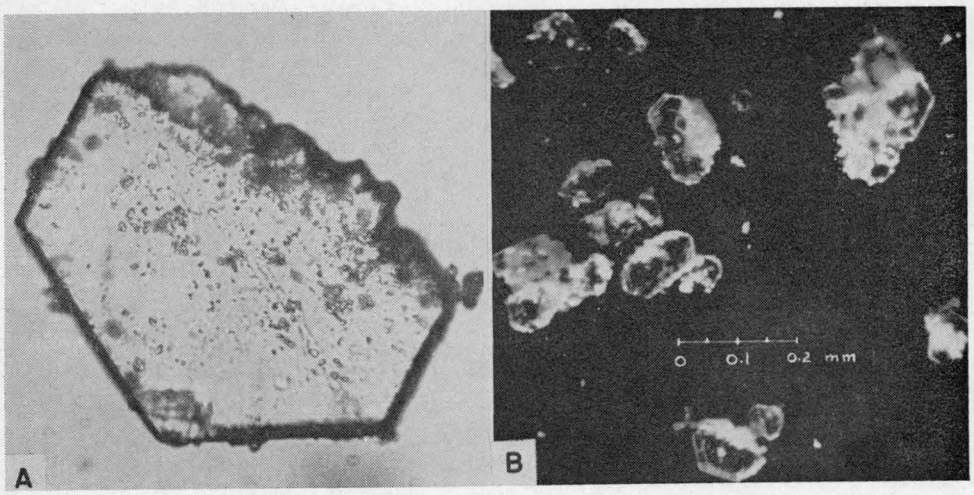


Fig. 2. Microscopic photographs of reaction product crystals.

## RESULTS

### Optical Properties

*Crystal selection.* A search was made with the binocular microscope for several of the largest perfect single crystals in the bentonite sample. These crystals were carefully removed from the matrix and were placed on a glass slide. Here they were cleaned of all adhering fragments by means of a single hair. They were then transferred to a second slide and a cover glass was placed over them for examination under the petrographic microscope. Doubly polarized light was used to determine which of the crystals selected were, in fact, perfect and free from inclusions, twinning, and intergrowths.

*Index of refraction.* Those crystals determined to be acceptable were transferred to individual slides for determination of the refractive index by the method of central illumination. The crystals were mounted in various immersion media of known refractive index and examined under the microscope. By bracketing the media used, one was found which coincided almost exactly with the refractive index of the crystal; that is, no reflection or refraction occurred at the boundary between the crystal and the immersion media. By this means the index of refraction of the ordinary ray was determined to be between 1.545 and 1.550. Because of the extreme thinness of the crystals, the index of refraction perpendicular to this direction was not determined exactly; however it was considerably less.

*Interference figure.* A crystal was mounted on a slide so that the basal

pinacoid was perpendicular to the axis of the scope and was viewed under convergent doubly polarized light. On inserting the Bertrand lens an axial cross appeared. Therefore, the crystal is uniaxial and must be either in the hexagonal or tetragonal system. No birefringence was observed with the crystal in this position. However, as soon as the pinacoidal face was inclined, marked birefringent colors appeared.

*Optic sign.* A gypsum plate was inserted in the slot above the objective lens. A lens shaped blue color appeared in the quadrants perpendicular to the direction of the slow ray in the plate thus determining the optic sign to be negative.

**Physical Properties**

*Geometry.* In plane polarized light the crystals were transparent and colorless, platy, and hexagonally shaped with beveling on the edges. A measurement of the hexagonal interior angles showed them to be exactly 60°.

*Specific gravity.* The heavy liquid method was used to determine the specific gravity of the crystals. In this method the crystal is immersed in a liquid of known density. If the crystal is denser than the liquid it will sink to the bottom; if less dense it will float. At some density of the liquid the crystal will neither sink nor float but will be suspended in the liquid. The density of the liquid and the crystal are then the same.

In this study mixtures of bromoform ($g = 2.890$) and carbon tetrachloride ($g = 1.595$) were varied to bracket the density of the crystal. Near the density of the crystals the mixture was varied in intervals of 0.005 gm/ml. A 5 ml sample was removed by a pipette at each point and weighed on an analytical balance to determine the exact density of the mixture. All of the crystals sank to the bottom at a liquid density of 2.060 and all were floating at 2.080 gm/ml; at intermediate densities crystals were observed in all three positions, floating, suspended, and on the bottom, establishing the specific gravity of the crystals at $2.07 \pm 0.01$.

**X-ray Investigations**

*Rotating crystal cylindrical camera.* A single crystal free from inclusions was selected and mounted with Ducot cement thinned with amyl acetate on a short fiber of glass wool in such a manner that the pinacoid was perpendicular to the axis of the glass wool fibre. The fibre was attached to a thin glass rod and the assembly was then inserted into the head of a two-circle goniometer which, in turn, was attached to the rotating spindle of the camera. The crystal was first aligned optically and then was brought into exact alignment by X-ray methods.

After alignment was completed, a fifteen hour exposure was taken. The diffracted rays are recorded on a cylindrical film whose axis is parallel and

concentric with the rotation axis. The spots on the film formed by the diffracted rays will produce a two dimensional pattern following a characteristic geometry (figure 3).

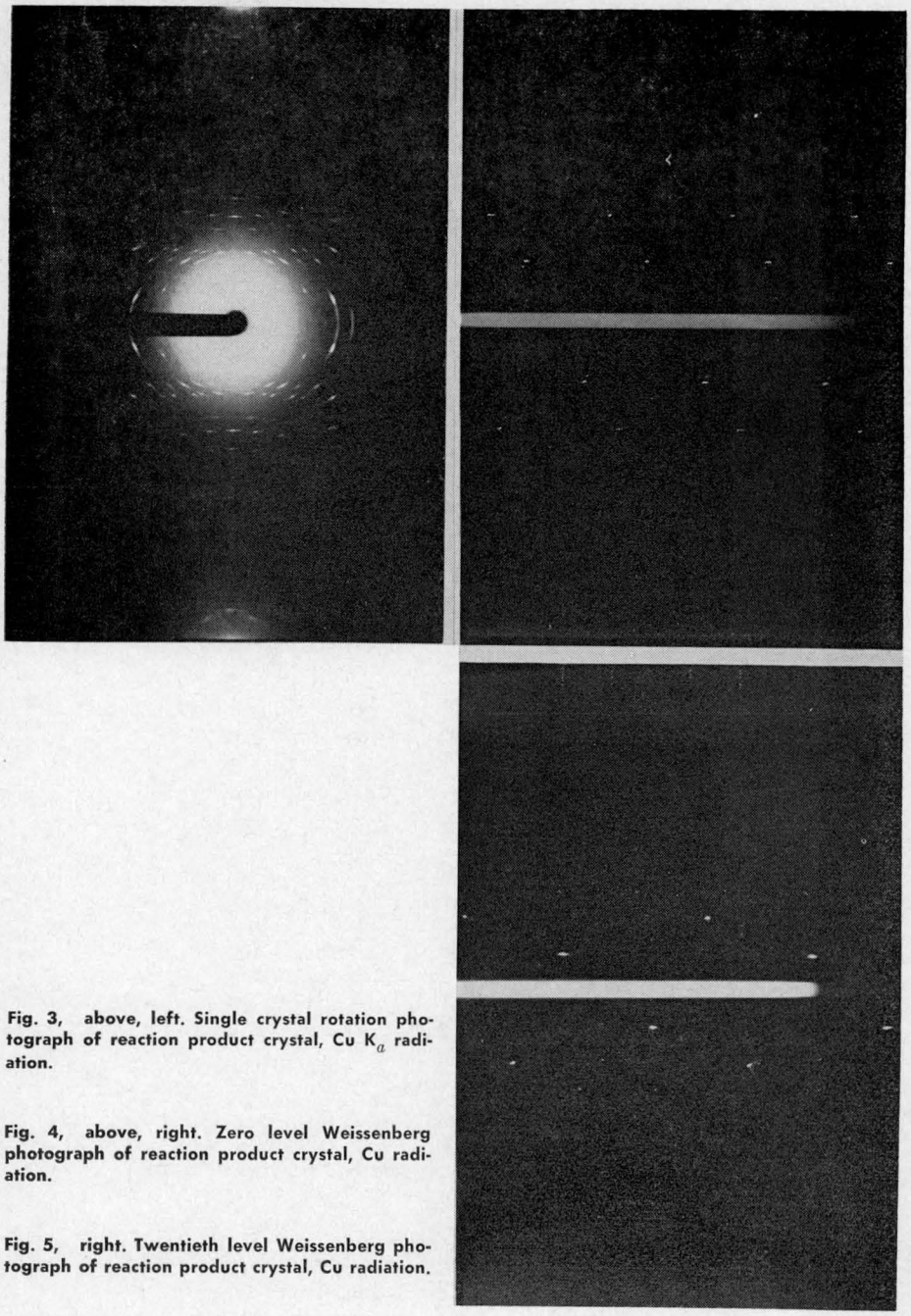

**Fig. 3, above, left. Single crystal rotation photograph of reaction product crystal, Cu $K_a$ radiation.**

**Fig. 4, above, right. Zero level Weissenberg photograph of reaction product crystal, Cu radiation.**

**Fig. 5, right. Twentieth level Weissenberg photograph of reaction product crystal, Cu radiation.**

The Bragg angle $\theta$ was found by measuring the $x$ and $y$ coordinates of each spot on the film and using the relationship

$$\text{Cos } 2\theta = \text{Cos } \tau \text{ Cos } \chi \qquad (1)$$

where $\tau = 2x$

and $\chi = \text{Tan}^{-1} \frac{2y}{D}$

where D = Camera diameter in millimetres

Then the interplanar spacing, d, is

$$d = \frac{\eta\lambda}{2 \text{ Sin } \theta} \qquad (2)$$

where $\lambda$ = Wave length of copper $K\alpha$ radiation.

The $d$ spacings and intensities for all spots observed on the film are tabulated in columns 1 and 2 of table III. Because of the size of these spots, these $d$ spacings are not as accurate as those found by other methods.

TABLE III. DIFFRACTION DATA, CU $K\alpha$ RADIATION

| Single x-tal rotation | | Hex. | Rhom. | Powder camera† | | Back Refl. Weissenberg |
|---|---|---|---|---|---|---|
| d | I | hk•l | hkl | d | I | d |
| 7.75 | 10 | 00.6 | 222 | 7.59 | 10 | |
| 3.83 | 7 | 01.8 | 332 | 3.85 | 7 | |
| 3.83 | 5 | 00.12 | 444 | | | |
| 3.44 | 4 | 10.10 | 433 | 3.42 | 4 | |
| 2.90 | 8 | 11.0 | 101 | 2.87 | 9 | |
| 2.86 | 1 | 11.3 | 210 | | | |
| 2.72 | 4 | 11.6 | 321 | 2.71 | 1 | |
| 2.54 | 6 | 10.16 | 655 | 2.52 | 6 | |
| 2.54 | 8 | 11.9 | 432 | | | |
| 2.50 | 1 | 20.2 | 200 | ..... | | |
| 2.46 | 7 | 02.4 | 220 | ..... | | |
| 2.34 | 9 | 11.12 | 543 | 2.33 | 6 | |
| 2.31 | 6 | 20.8 | 422 | 2.30 | 8 | |
| 2.22 | 6 | 02.10 | 442 | 2.20 | 4 | |
| 2.14 | 10 | 01.20 | 776 | 2.11 | 5 | |
| 2.13 | 5 | 11.15 | 654 | | | |
| 2.08 | 1 | 12.11 | 542 | ..... | | |
| 2.01 | 6 | 20.14 | 644 | 2.07 | 1 | |
| 1.96 | 1 | 10.22 | 877 | ..... | | |
| 1.94 | 5 | 11.18 | 765 | 1.93 | 3 | |
| 1.92 | 4 | 02.16 | 664 | | | |
| 1.88 | 4 | 21.4 | 310 | 1.86 | 3 | |
| 1.87 | 4 | 12.5 | 320 | | | |
| 1.81 | 3 | 12.8 | 431 | 1.79 | 1 | |
| 1.76 | 1 | 21.10 | 532 | ..... | | |
| 1.73 | 1 | 21.22 | 976 | ..... | | |
| 1.71 | 9 | 20.20 | 866 | 1.72 | 4 | |
| 1.69 | 3 | 21.13 | 655 | ..... | | |
| 1.68 | 9 | 30.0 | 112 | 1.66 | 5 | 1.706 |
| 1.66 | 2 | 12.14 | 653 | | | |

TABLE III. (CONTINUED)

| Single x-tal rotation d | I | Hex. hk•l | Rhom. hkl | Powder camera† d | I | Back Refl. Weissenberg d |
|---|---|---|---|---|---|---|
| 1.64 | 1 | 11.24 | 987 | 1.63 | 4 | |
| 1.64 | 7 | 30.6 | 411 | | | |
| 1.594 | 3 | 21.16 | 754 | ..... | | |
| 1.54 | 5 | 30.12 | 633 | 1.53 | 3 | |
| 1.483 | 6 | 12.20 | 875 | 1.475 | 4 | |
| 1.464 | 3 | 20.26 | 1088 | ..... | | |
| 1.45 | 6 | 22.0 | 202 | 1.438 | 1 | 1.465 |
| 1.43 | 4 | 22.6 | 420 | 1.416 | 1 | |
| 1.396 | 2 | 13.1 | 242 | ..... | | |
| 1.38 | 1 | 31.5 | 410 | ..... | | |
| 1.36 | 4 | 22.12 | 642 | 1.355 | 2 | |
| 1.301 | 3 | 12.26 | 1097 | 1.303 | 2 | |
| 1.279 | 1 | 13.16 | 763 | ..... | | |
| 1.229 | 1 | 04.8 | 440 | | | |
| 1.200 | 2 | 31.20 | 965 | 1.195 | 1 | |
| 1.166 | | 13.22 | 985 | 1.166 | 1 | |
| 1.131 | 1 | 23.8 | 530 | 1.137 | 1 | |
| 1.121 | 2 | 32.10 | 731 | ..... | | |
| 1.109 | 1 | 04.20 | 884 | ..... | | |
| 1.097 | 5 | 41.0 | 213 | 1.089 | 1 | 1.094 |
| 1.092 | 3 | 23.14 | 752 | | | |
| 1.088 | 3 | 41.6 | 510 | 1.081 | 1 | |
| 1.072 | 1 | 32.16 | 870 | ..... | | |
| 1.056 | 4 | 41.12 | 732 | 1.050 | 1 | |
| 1.034 | 3 | 23.20 | 974 | 1.029 | 1 | |
| 0.984 | 1 | 05.10 | 810 | ..... | | |
| 0.967 | 3 | 33.0 | 303 | ..... | | 0.963 |
| 0.960 | 1 | 50.14 | 833 | ..... | | |
| 0.958 | 2 | 33.6 | 521 | ..... | | |
| 0.937 | 1 | 33.12 | 741 | 0.933 | 1 | |
| 0.926 | 2 | 32.4 | 411 | ..... | | |
| 0.923 | 2 | 50.20 | 1055 | ..... | | |
| 0.876 | 2 | 42.20 | 1064 | ..... | | |
| 0.843 | 2 | 60.6 | 600 | ..... | | |
| 0.841 | 2 | 15.20 | 983 | ..... | | |
| 0.836 | 2 | 60.0 | 224 | ..... | | 0.8314 |
| 0.818 | 2 | 52.12 | 831 | ..... | | |
| 0.803 | 5 | 52.0 | 314 | ..... | | 0.7988 * |

*Other hexagonal indices observed on Weissenberg photographs in addition to the one listed, x0z, when z + 3n, are $\bar{x}xz$, $0\bar{x}z$, $0x\bar{z}$, $\bar{x}0\bar{z}$, and $x\bar{x}\bar{z}$.

Other indices observed in addition to 0yz when z + 3n are $\bar{y}0z$, $y\bar{y}z$, $y0\bar{z}$, $\bar{y}yz$, and $0\bar{y}\bar{z}$.

Other indices observed in addition to xyz when z + 3n are $\bar{x}$,x+y,z; $\bar{x}+\bar{y}$, x, z; $\bar{y},\bar{x},z$; y,$\bar{x}+\bar{y}$,z; x+y, $\bar{y}$,z; y,x,$\bar{z}$; $\bar{y}$,x+y, $\bar{z}$; $\bar{x}+\bar{y}$, y, z; $\bar{x},\bar{y},z$; x,$\bar{x}+y,z$; x+y,$\bar{x},\bar{z}$.

All possible indices observed when z = 3n.

†In addition to the powder camera data indicated in the table, lines were also observed at 8.29, 5.52, 4.45, and 3.16 Ångstroms which were identified as montmorillonite.

## Weissenberg Method

The rotating crystal method has several rather serious limitations. A

superposition of several reflections occurs causing difficulty in unequivocally indexing the reflections and determining their intensities. The Weissenberg method solves this difficulty by translating the film in a direction parallel to the rotation axis at a rate proportional to the angular rotation of the crystal. So that only one layer line at a time will be recorded, the diffraction cone of that layer is isolated by surrounding the crystal with a closed hollow metal cylinder containing a circular slit through which the desired layer line cone is permitted to emerge.

The equi-inclination Weissenberg method was used in which

$$\mu = -\nu$$

where $\mu =$ the complement of the angle between the direct beam and the lattice rows

and $\nu =$ the complement of the semi-opening angle of the diffracted cone

In this method any central layer line projects on the n - level Weissenberg photograph as a straight line of slope 2 since this method brings the crystal rotation axis exactly on the circumference of the reflecting circle. Non-central lattice lines project as ovals, the ovals assuming the same geometric pattern for each level. By combining two of these patterns separated 90° from each other, a template may be constructed permitting indexing of the reflections directly.

**Indexing of reflections**

Weissenberg photographs were taken of each layer line from 0 to 26. Prints of the zero level and the twentieth level are reproduced (figures 4, 5). Each of the twenty-six levels were indexed by means of the template discussed earlier, and the results are tabulated in column 3 of table III.

Since the central layer lines occurred at sixty degree intervals, the crystal system was confirmed as being hexagonal. Also by noting the Weissenberg projections of chains of diamond shaped cells which occur along the position symmetry lines of the Weissenberg photographs, it is obvious that the crystals are in the rhombohedral division of the hexagonal system.

To convert the hexagonal indices given in column 3 of table III to rhombohedral indices, the relationship given in equations 4, 5, & 6 were used.

$$h_r = \tfrac{1}{3}\,(2\,h + k + l) \tag{4}$$

$$k_r = \tfrac{1}{3}\,(-\,h + k + l) \tag{5}$$

$$l_r = \tfrac{1}{3}\,(-\,h - 2k + l) \tag{6}$$

The results of this conversion appear in column 4 of table III.

**Hexagonal cell lattice constants**

To determine precise lattice constants, chromium $K\alpha$ radiation was used to move some of the lines observed with copper radiation into the back reflection region of the powder camera film. A fine collimator (0.7 mm) was

used with helium atmosphere and Straumanis film loading. Temperature during the exposure was maintained at 29° C.

The $a_o$ and $c_o$ constants were computed from the measured d-spacings corrected for film shrinkage and were extrapolated against the function

$$\frac{\text{Cos}^2\,\theta}{\text{Sin}\,\theta} + \frac{\text{Cos}^2\,\theta}{\theta} \tag{7}$$

Table IV gives the $a_o$ and $c_o$ constants for the reflections listed which represent results of the ninth a/c approximation and recalculation. Since there was no slope to the $\theta$ plot, the average value of the computed constants may be used.

TABLE IV. HEXAGONAL LATTIC CONSTANTS, CR Kα RADIATION

| hkl | $a_o$ | hkl | $c_o$ |
|---|---|---|---|
| 30.0 | 5.7549 | 20.20 | 46.659 |
| 22.0 | 5.7552 | 21.16 | 46.689 |
| 22.6 | 5.7549 | 30.12 | 46.657 |
| | Av. 5.7550 | 12.20 | 46.611 |
| | | 20.26 | 46.687 |
| | | 12.26 | 46.640 |
| | | 31.20 | 46.663 |
| | a/c = 0.12335 | | 46.627 |
| | | | Av. 46.654 |

The standard deviation of the above values is 0.025 which gives 90% confidence limits for $c_o$ of $46.654 \pm 0.018$.

A further check on the $a_o$ lattice constant was made by the back reflection Weissenberg method, data for which appears in column 7 of table III. Extrapolation of this data against the $\theta$ function, equation 7, gives an $a_o = 5.756$ Å. Since the line breadth on the powder camera film is finer than that of the spots on the Weissenberg film, more accurate determinations are possible utilizing the powder camera.

A rough check on the $c_o$ spacing is possible by utilizing the single crystal rotation photograph. The layer line spacing, $\zeta$, of the reciprocal lattice is given by

$$\zeta_n = \frac{y_n}{\sqrt{r_f^2 + y^2}} \tag{8}$$

where $r_f$ = film radius

and $y_n$ = dist. from film center to any given layer line.

To find the identity period along the rotation axis (c) of the crystal, equation (9) is used.

$$c_o = \frac{n\lambda}{\zeta_n} \tag{9}$$

The average of the $\zeta$'s for the first sixteen layer lines was used, and a value for $c_o$ of 46.72 Å was computed. Since the film shrinkage could not be computed for this film the $c_o$ spacing compares favorably with that shown in table IV.

**Hexagonal cell volume**

The volume of the hexagonal unit cell is given by

$$V = \tfrac{1}{2}\sqrt{3\, a^2\, c.} \tag{10}$$

Since $a_o = 5.7550$ Å and $c_o = 46.654$ Å, then the volume is 1338.17 Å$^3$ or $133.817 \cdot 10^{-23}$ cm$^3$.

**Rhombohedral cell lattice constants**

Designating the hexagonal lattice constants as $a_H$ and $c_H$, the corresponding rhombohedral constants are

$$\partial_R = \tfrac{1}{3}\sqrt{3a^2_H + c^2_H} \tag{11}$$

$$\mathrm{Sin}\frac{\partial}{2} = \frac{3a_H}{2\sqrt{3a^2_H + c^2_H}}$$

and $a_R = 15.902$Å and $\alpha = 20.850°$. (12)

**Space group determination**

It has already been shown that the crystal belongs to the rhombohedral division of the hexagonal system. Other information is also available from the Weissenberg photographs to help in the space group determination.

By comparing the zero level photograph of figure 4 (and all other 3n levels) with figure 236 in Buerger[4] it is apparent that these levels have the characteristic appearance of plane point group $C_{6l}$. On the original films even more so than on the prints, the intensities of spots at the same height about a central line can be seen to be equal. The films of all other levels show the same relationship of the intensities; however, on levels other than 3n, the plane point group is $C_{3l}$ since the period is 120°. This combination has been identified as belonging to centrosymmetrical crystal class $D_{3d}$[4, p. 474].

With this information the possible space groups to which the crystal may belong may be determined by observing any systematic absences of reflections.

No systematic absences were observed in the general hk*l* reflections for the rhombohedral cell. In the hh*l* reflections, the only *l* indices occurring

were for those in which $l = 2n$, indicating a (110) glide plane of component $\frac{c}{2}$ (c-glide). In the hoo, oko and oo*l*, h, k, and *l*, respectively, were equal to 2n indicating (100), (010), and (001) screw axes of components $\frac{a_1}{2}$, $\frac{a_2}{2}$, and $\frac{a_3}{2}$.

Unfortunately, X-ray photographs cannot distinguish between the presence or absence of a center of symmetry. Thus, although it is known that a three-fold axis is present, it is not possible to determine whether it is a rotation or an inversion axis.

Still, since it is known that the crystal has a three-fold axis, is rhombohedral, has a c-glide, and belongs to the centrosymmetrical class $D_{3d}$, the space group then must be either $D^6_{3d}$ (R3c) or $C^6_{3v}$ (R3c).

## CHEMICAL PROPERTIES

### Solubility

The crystal is soluble in most dilute acids including 0.05 normal hydrochloric acid, sulfuric acid, and acetic acid.

### Chemical Analysis

The total amount of crystals separated during the course of this investigation was about 8 mg. Although the crystals were produced in abundance, the difficulty of separating them from the matrix accounts for the small amount of pure crystals isolated. Even with these relatively pure crystals, it was exceedingly difficult to remove completely all the montmorrillonite which clung to them with great tenacity. Therefore a good quantitative analysis was not obtained. Qualitatively it was determined that calcium, aluminum, silicon, and a very small amount of sodium were present; iron was absent.

### X-ray analysis

Comparison of the observed *d* spacings and intensities with those in the X-ray powder data file[2] showed a striking resemblance between the diffraction data of this crystal and the data for two calcium aluminate hydrates: $4CaO{\cdot}Al_2O_3{\cdot}xH_2O$ and $3CaO{\cdot}Al_2O_3{\cdot}8\text{-}12H_2O$. However, small variances in *d* spacings and intensities do not permit positive identification with either of these two compounds. Wells[13] reported *d* spacings for $4\ C_aO{\cdot}Al_2O_3{\cdot}13H_2O$ which compared almost exactly, except for the basal reflection, with those tabulated in table III for this crystal. However, intensity and

indexing data differ considerably.

Others have interpreted the appearance of reflections at 7.8, 3,82 and 2.87 Å to indicate the presence of $C_4A \cdot 13H_2O$[5].

The index of refraction of the ordinary ray of these crystals is the same as that observed for compounds opproaching the formula $C_4A \cdot 13H_2O$[13]. This compound was also uniaxial negative.

However, the silicon observed in the qualitative analysis of the crystals leaves some doubt as to the exact formula. Certainly, from the observed $d$ spacings, the structure of the crystal must be almost isostructural with the tetracalcium aluminate hydrates discussed above. Deviations in intensities may be due to incorporation of silica in the crystal structure or due to $CO_2$ in solid solution in the crystal.

On the basis of unit cell volume and density the $C_4A \cdot 13H_2O$ hypothesized formula would indicate a unit cell composed of 3 molecules since

$$N = \frac{\rho V A}{M} \tag{13}$$

where N = No. of molecules/unit cell
$\rho$ = density (2.07 gms/cm$^3$)
V = unit cell volume (133.82—10$^{-23}$ cm$^3$)
A = Avogadro's number (6.025•10$^{23}$ Molecules/mol)
and M = formula weight (560.49 gm/mole).

Then N = 2.97 $\approx$ 3 molecules per unit cell.

**Future investigations**

The prime objective of future research will be to produce a larger amount of these crystals and completely separate them from all contaminants to obtain an exact quantitative analysis and definitely establish their chemical composition.

## CONCLUSIONS

1. In mixtures of lime, soil M-67, and water which are moist cured for thirty days, a crystalline reaction product develops. This product is produced in optimum quantities at lime contents of 20 percent by dry weight of soil and at high moisture contents. Only the clay size portion of the soil enters into the reaction.

2. The same crystalline reaction product develops in mixes of lime, water and bentonite, but only after a considerably longer curing time is it produced in considerable quantity.

3. The crystals produced are transparent, colorless, platy, and hexagonally shaped. Their density is 2.07 $\pm$ .01 gm/cm$^3$ at 70° F. Observations under the petrographic microscope prove them to be uniaxial negative with

$\omega = 1.548 \pm .002$.

4. X-ray investigations of the crystal structure prove that it is the rhombohedral division of the hexagonal system in space groups $D^6_{3d}$ ($R\bar{3}c$) or $C^6_{3v}$ (R3c). Intensities from powder camera films and indexing by Weissenberg methods establish the four strongest lines at 7.59 Å, 00.6; 2.87 Å, 110; 2.30 Å, 20.8; and 3.85 Å, 01.8 and 00.12. Lattice constants are $a_o = 5.7550$ Å and $C_o = 46.654$ Å for the hexagonal cell and $a = 15.902$ Å and $d = 20.850°$ for the rhombohedral cell.

5. The chemical composition of the crystal could not be precisely determined; however, the *d* spacings indicate a structure similar to that for $4Ca\ O.Al_2O_3\ .13H_2O$ and it is possible that this is the composition of this crystal. If so, previously recorded data for indexing and unit cell dimensions are in error and should be revised.

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# RELATION OF STRENGTH TO COMPOSITION AND DENSITY OF LIME TREATED CLAYEY SOILS

by

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(Highway Research Board Proceedings. 1961.)

## ABSTRACT

The effects of dolomitic monohydrate "type N" and calcitic hydrated limes, Standard and Modified AASHO density compaction, and the predominant type of clay mineral in the soil on the immersed strength of soil lime mixtures are examined.

Dolomitic lime gave higher strengths to montmorillonite and illite clay soils, but gave higher strengths only to some kaolinite clay soils. This trend held at both compactive energies. Modified density compaction was found to give significantly higher strengths than standard density compaction.

## INTRODUCTION

Small additions of lime to clayey soils may improve consistency limits, workability and ease of pulverization, and volume change characteristics of such soils, and use of additional amounts of lime may contribute to strength increases[4]. However, much more information is needed on the relations of these property improvements to such variables as lime and soil composition and to compacted density. The purpose of this paper is to present some experimental findings concerning the relation of cured strength of lime treated soil mixtures to predominant soil clay mineral, type and amount of lime, and compacted density.

## THE MATERIALS USED

### Soils

Nine soils from various parts of the United States were used in the investigation (table I). The major groups of soil clay minerals were represented in the clay fractions of these soils; three (AR-2, AR-3, AR-7) were dominated by montmorillonite, three (AR-4, AR-8, AR-9) by illite, and

TABLE I. SOIL SITE CHARACTERISTICS

| Soil* designation | Sampling location | Geological description | Soil series and horizon | Sampling depth, inches |
|---|---|---|---|---|
| AR-2 | Ringgold County, Iowa | Kansan-age glacial till calcareous | Sherby (Burchard), C horizon | 54-126 |
| AR-3 | Harris County, Texas | Coastal Plain deposit, largely deltaic, calcareous | Lake Charles, probably C horizon | 39-144 |
| AR-7 | Keokuk County, Iowa | Plastic loess, Wisconsin age, noncalcareous | Mahaska, C horizon | 36-77 |
| AR-4 | Monroe County, Mich. | Probably Wisconsin-age glacial till, calcareous | Unknown, C horizon | Unknown |
| AR-8 | Livingston County, Ill. | Wisconsin-age glacial till, calcareous | Clarence, C horizon | 46-56 |
| AR-9† | Goose Lake region, Ill. | Commercial product, noncalcareous | Unknown, probably C horizon | Unknown |
| AR-5 | Orange, Va. | Residual soil over diorite, noncalcareous | Davidson, B horizon | Unknown |
| AR-6 | Durham County, N. C. | Residual soil over medium grained biotite granite, noncalcareous | Durham, B horizon | 24 below A horizon |
| AR-10‡ | N. C. | Unknown, noncalcareous | Unknown, probably C horizon | Unknown |

*Iowa Engineering Experiment Station Soil Research Laboratory Sample Designation.
†AR-9 is the commercial product Grundite, supplied by the Illinois Clay Products Company, 214 Barber Building, Joliet, Illinois.
‡AR-10 was supplied by the Harris Clay Company, Spruce Pine, North Carolina.

three (AR-5, AR-6, AR-10) by kaolinite group clay minerals (table II). The montmorillonite clay soils contained some illite. One of the illite clay soils, AR-4, had an appreciable amount of chlorite. The clay fraction of soil AR-5 was rich in halloysite, a kaolinite subgroup mineral. Soils AR-6 and AR-10 had substantial amounts of mica.

### Limes

Six commercial limes were used, three calcitic hydrated limes and three dolomitic monohydrate "Type N" limes (Table III). Limes were used within six months of their receipt from manufacturers. When not in use lime containers were sealed tightly to prevent carbonization of the lime.

TABLE II. PROPERTIES OF SOILS

| Soil designation | AR-2 | AR-3 | AR-7 | AR-4 | AR-8 | AR-9 | AR-5 | AR-6 | AR-10 |
|---|---|---|---|---|---|---|---|---|---|
| Textural composition,* % | | | | | | | | | |
| Gravel† (> 2 mm.) | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Sand (2-0.074 mm.) | 20.9 | 3.0 | 0.3 | 7.0 | 10.0 | 6.4 | 11.0 | 45.3 | 38.4 |
| Silt (0.074-0.005 mm.) | 40.6 | 36.0 | 60.8 | 36.0 | 38.0 | 18.6 | 37.0 | 18.3 | 34.4 |
| Clay (< 0.005 mm.) | 38.5 | 61.0 | 39.0 | 57.0 | 52.0 | 75.0 | 42.0 | 36.5 | 7.0 |
| Clay (< 0.002 mm.) | 33.0 | 51.0 | .... | 44.0 | 41.0 | 59.3 | 29.5 | 30.0 | 4.0 |
| Passing No. 10 sieve | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Passing No. 40 sieve | 100.0 | 99.0 | 100.0 | 98.0 | 96.0 | 99.9 | 90.0 | 67.0 | 64.8 |
| Atterberg limits,‡ % | | | | | | | | | |
| Liquid limits, % | 41.2 | 64.6 | 52.1 | 44.0 | 35.5 | 54.8 | 43.5 | 51.0 | 43.0 |
| Plastic limit | 16.7 | 17.6 | 20.0 | 21.1 | 17.5 | 27.1 | 27.0 | 25.5 | N.P. |
| Plasticity index | 34.5 | 47.0 | 32.1 | 22.9 | 18.0 | 27.7 | 16.5 | 25.5 | N.P. |
| Chemical | | | | | | | | | |
| pH§ | 8.5 | 8.8 | 5.6 | 8.4 | 8.3 | 5.5 | 5.9 | 5.7 | 5.5 |
| C.E.C.,¶ m.e./100 g | 17.5 | 27.3 | 23.5 | 14.5 | 10.8 | 19.1 | 11.0 | 8.4 | 4.6 |
| Carbonates,£ % | 7.4 | 16.6 | 1.5 | 7.2 | 22.5 | 1.92 | 0.65 | 0.1 | 0.07 |
| Organic matter,** % | 0.06 | 0.13 | 0.2 | 0.64 | 0.7 | 1.54 | 2.62 | 0.1 | 0.02 |
| Predominant clay mineral†† | M | M | M | I and C | I | I‡‡ | H | K | K |
| Classification | clay | clay | silty | clay | clay | clay | clay | clay | sandy |
| Textural§§ | | | clay | | | | | | loam |
| AASHO¶¶ | A-7- | A-7- | A-7- | A-7- | A-6 | A-7- | A-7- | A-7- | A-5 |
| | 6(14) | 6(20) | 6(18) | 6(14) | (11) | 6(18) | 6(12) | 6(11) | (1) |

* ASTM Method D 423-54T[2].

† Textural gradation tests were performed only on the soil fraction passing the No. 10 sieve. All soils used contained less than 5% gravel.

‡ ASTM Methods D 423-54T and D 424-54T[2].

§ Glass electrode method using suspension of 15 g. soil in 30 cc. distilled water.

¶ Ammonium acetate (pH = 7) method on soil fraction < 2 mm. (No. 10 sieve).

£ Versenate method for total calcium.

**Potassium bichromate method.

††X-ray diffraction analysis method. Symbols mean: M, Montmorillonite; I, illite; I and C, illite and chlorite; H, halloysite (kaolinite group mineral); K, kaolinite.

‡‡By analysis of chemical constituents furnished by manufacturer, assuming all alkalies as potassium and determining the number of potassium ions per unit cell.

§§From the triangular chart developed by the U. S. Bureau of Public Roads, but 0.074 mm. was used as the lower limit of the sand fraction[7].

¶¶AASHO Designation: M 145-49[1].

## Water

Distilled water (pH = 6 to 7) was used in all tests.

## OPERATIONAL PROCEDURES

### Soil preparation

An identical procedure was used in preparing each soil for all tests. As the soil was received it was spread out to air dry on brown wrapping paper placed on a concrete table. After a few days the soil was hand crushed, if necessary, and sieved through a No. 10 sieve. Material retained on the sieve was then placed in a steel bowl (mortar) for crushing. The crusher was a drill press on which was mounted a rubber pestle. A free sliding metal disk,

the size of the top of the bowl, was mounted on the pestle stem to prevent loss of soil fines during crushing. The soil was crushed and sieved until the soil aggregations were completely broken down. Particles that would not pass the No. 10 sieve were discarded. Soil passing the No. 10 sieve was mixed to obtain uniformity and placed in 30 gallon galvanized cans until used.

TABLE III. PROPERTIES AND PRODUCTION INFORMATION OF HYDRATED LIMES

| | Calcitic hydrated lime | | | Monohydrate "Type N" dolomitic lime | | |
|---|---|---|---|---|---|---|
| | C-US* | C-M | C-L | D-US | D-M | D-W |
| Chemical constituent, % by wt† | | | | | | |
| Calcium oxide, CaO | 73.0 | 73.46 | 73.9‡ | 49.1 | 47.52 | 48.3 |
| Calcium hydroxide, $Ca(OH)_2$ | 96.5§ | 97.04 | 97.68 | 64.8§ | 61.81 | 63.7§ |
| Magnesium oxide, MgO | 0.3 | 0.93 | 0.64 | 32.0 | 33.50 | 33.2 |
| Silica, $SiO_2$ | 0.6 | 0.34 | 0.69 | 0.4 | 0.6 | 0.6 |
| Iron-Alumina, $R_2O_3$ | 0.3 | 0.24 | 0.59 | 0.3 | 0.62 | 1.1 |
| Total loss on ignition | 24.5-27.0 | 24.92 | 24.22 | 17.0 | 17.83 | 16.8 |
| Ca:Mf ratio | .... | .... | .... | 1.815:1¶ | 1.682:1¶ | 1.732:1¶ |
| Trade name | Kemikal | .... | Chemical Lime | Kemidol | .... | Limate |
| Processing plant | New Braunfels, Texas | Hannibal, Missouri | .... | Genoa, Ohio | Thornton, Illinois | .... |

*Manufacturer by lime code designation:
D-US and C-US, U. S. Gypsum Co., 300 W. Adams St., Chicago 6, Illinois.
D-M and C-M, Marblehead Lime Co., 300 West Washington Street, Chicago 6, Illinois;
C-L, Linwood Stone Products Co., Inc., R.R. 2, Davenport, Iowa;
D-W, Western Lime and Cement Co., Box 2076, Milwaukee 1, Wisconsin.
†Data supplied by company concerned.
‡Calculated by molecular weight ratios from amount of $Ca(OH)_2$ present.
§Calculated by molecular weight ratios from amount of CaO present.
¶Calculated as ratio of calcium to magnesium by weight from amount of materials present as their oxides.

## Mixing

A predetermined amount of air dry soil was weighed out on a balance sensitive to 0.1 grams and was placed in a mixing bowl. Lime additive, if used, was weighed and hand mixed with the soil. Additional dry mixing was for 1 minute with a Hobart, Model C-100 ¼ H.P. mixer, at low speed. Distilled water was added, and the mixture was mixed for 2 minutes. The mixture was then thoroughly stirred by hand to insure that no materials were left unmixed on the sides and bottom of the bowl. The mixture was mixed again for 30 seconds to complete the process.

## Molding

Test specimens were prepared by use of the Iowa State University molding apparatus[3, 8]. The 5 pound hammer, called the standard hammer,

is used to compact a predetermined amount of soil mixture in a 2 inch diameter mold to a density near Standard AASHO density (AASHO Designation: T99-57)[1]. The 10 pound hammer, called the modified hammer, is used to compact a predetermined amount of soil in a 2 inch diameter mold to a density near Modified AASHO density (AASHO Designation: T180-57)[1].

After the material was mixed it was covered with a damp cloth to prevent evaporation. When test specimens were prepared, a predetermined amount of the mixture was placed in the compaction mold. The proper hammer was used to attain the desired density. The resultant soil cylinder was extruded from the mold with an hydraulic jack. The compacted specimen was weighed to the nearest 0.1 gram and the height was measured to the nearest 0.001 inch. The height of the specimen was required to be 2.000 inches $\pm$ 0.050 inch. All specimens not within these limits were rejected.

### Curing

Immediately after it was weighed and measured, the specimen was wrapped in waxed paper and sealed with cellulose tape to prevent loss of moisture and carbonization of lime from carbon dioxide in the air. The wrapped specimens were placed on shelves in a curing room where the relative humidity was at least 90% and the temperature was 75° F $\pm$ 6°.

### Testing

The apparatus used for testing the strength of the specimens was a Model AP-170 Stability Testing Hachine driven by a ½ H.P. motor with belt reduction. Loads are indicated on a sensitive, 10,000 pound capacity proving ring which is supplied with a dial indicator reading to 0.0001 inch deflection. Strain was applied to the test specimen at a constant rate of 0.1 inch per minute. Strain on the proving ring is related to load by means of a calibration chart.

At the time of testing, specimens were removed from the curing room, unwrapped, and immersed in distilled water for 24 hours $\pm$ 1 hour. Then they were tested to failure to determine the unconfined compressive strengths. Three identical specimens of each mixture were always tested, and the strengths reported are generally the average of three specimens. If the strength of one specimen of a set fell out of the range of 10% of the average strength $\pm$ 3 pounds per square inch, the other two samples supplied the average.

## EXPERIMENTAL WORK PROCEDURES

### Preliminary study

A series of moisture density and moisture strength relationship tests were conducted on mixtures of each soil and 4, 8 and 12% of each lime to

evaluate and compare the optimum moisture contents for maximum dry density and maximum strength. These relationships were established for each compactive energy and each mixture by molding five sets of three specimens each at different determined moisture contents. After each specimen was weighed and measured, it was moist cured for 7 days, immersed in water for 24 hours, and then tested for strength.

Graphs of dry density versus molding moisture content and of strength versus molding moisture content were plotted. A smooth curve was drawn connecting plotted points and the optimum moisture contents were extracted from the graphs. Optimum moisture contents of 6 and 10% lime mixtures were determined by straight line interpolations.

Although the optimum moisture contents for maximum dry density and maximum strength were not always identical for each mixture studied, they were nearly the same in most, and it seemed best to use optimum moisture content for maximum dry density as the molding moisture content for the mixtures evaluated in the study of strength versus lime content. This decision applied to the preparation of specimens at both Standard and Modified densities.

With few exceptions, the soils molded with Standard AASHO density compaction had their optimum moisture content for maximum dry density increased 1.7 to 6.8% by the lime treatments, and their maximum dry density was lowered 2.5 to 17.4 pcf. With the same lime treatments, but with Modified AASHO density compaction, the optimum moisture content for maximum dry density increased 0.5 to 3.4% and the maximum dry density decreased 3.7 to 11.9 pcf.

The kind of lime used had a significant effect on the optimum moisture contents for maximum dry density and maximum strength. For treatments of a soil with equal amounts of different limes, the deviations among optimum moisture contents often exceeded 2%. Generally, however, the influence of kind of lime on the optimum moisture contents was least for mixtures at Modified density. Except with one soil, AR-6 at Modified density, dolomitic limes produced higher maximum dry densities than calcitic limes.

**Strength versus lime content.**

Specimens of the nine soils were molded at 4, 6, 8, 10 and 12% lime, by dry weight of soil used, with both U. S. Gypsum calcitic and "Type N" dolomitic limes. The mixtures were molded at densities near Standard and Modified AASHO densities.

Six specimens at each density were molded from each mixture. The moisture content used was the optimum moisture content for maximum dry density for the particular soil, lime content, type lime, and compactive effort used, as determined by the preliminary study. Moisture content samples were taken at the conclusion of mixing of each batch and again after the last specimen of the batch was molded. The average moisture content

for the two samples taken was required to be within plus or minus 1 percent of the optimum moisture content specified. Specimens 1, 3, 5 of each set were cured 7 days and specimens 2, 4, and 6 were cured 28 days.

After curing and immersion the samples were tested. Using average strength values for each three-specimen set, curves were constructed depicting strength versus lime content for each soil at the additional parameters of type of lime and compactive effort.

As a check to insure that the strengths obtained were indicative of type of lime used and not of one special lime, four other limes were used, two calcitic and two "Type N" dolomitic, with selected soils: Montmorillonite clay soil, AR-2; illite clay soil, AR-8, and kaolinite clay soil, AR-6, were used. Mixtures were molded to near Standard AASHO density. Relative values obtained in this study could then be compared to those obtained in the main study. Using average strength values for each three-specimen set, curves were constructed depicting strength versus lime content for each lime at the additional parameter of soil type.

## RESULTS

### Montmorillonite clay soils

At both Standard and Modified AASHO density, 7 and 28 day cured unconfined compressive strengths of the three montmorillonite clay soils (AR-2, AR-3, AR-7) at lime contents at or greater than 6% were significantly higher for U. S. Gypsum dolomitic lime than for U. S. Gypsum calcitic lime; 150 to 250 psi higher at Standard density, and 150 to 300 psi higher at Modified density (figure 1). Mixtures of soil AR-2 and each of different limes, two dolomitic and two calcitic, also showed that dolomitic lime gives highest strengths (figure 2).

Soil lime mixtures compacted at Modified density attained much higher strengths than when compacted at Standard density, for example, 200 to 350 psi higher for dolomitic lime mixtures and 200 to 250 psi higher for calcitic lime mixtures (figure 1).

There is probably an optimum lime content for maximum strength which varies for each soil[5, 6]. If optimum lime content is taken to imply a strength maximum or a greatly decreased rate of strength gain with increasing lime content, the optimum dolomitic lime content for maximum strength is at or over 6%. And the optimum calcitic lime content is at or probably less than 4% (figure 1).

### Illite clay soils

At Standard density lime treated illite clay soils (AR-4, AR-8, AR-9) did not develop high strengths; at best the strengths were only between 100 and 300 psi after 28 days of curing (figure 1). However, at lime con-

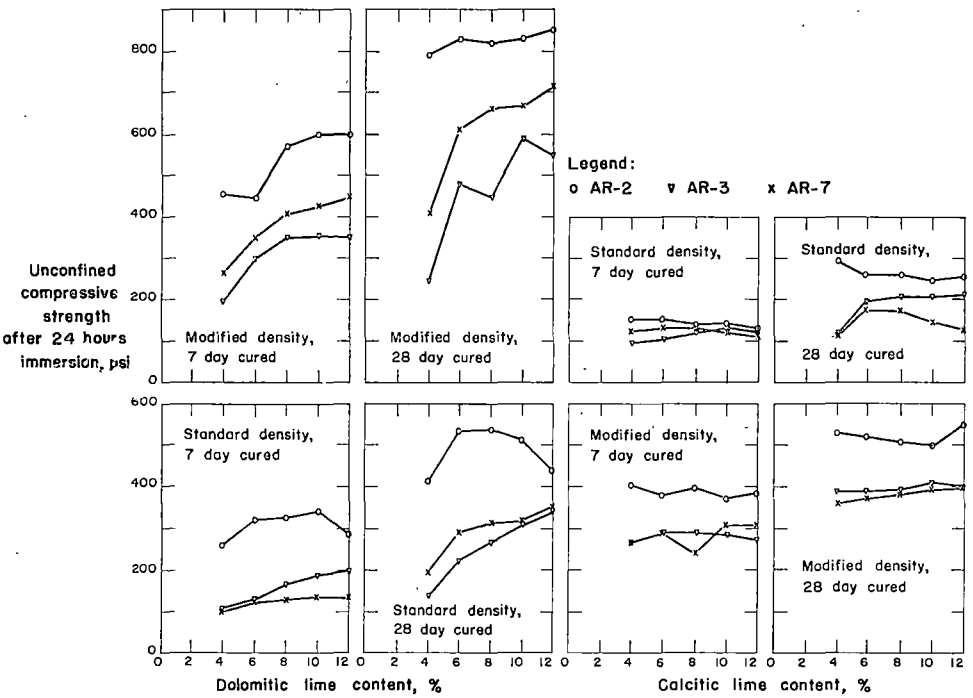


**Fig. 1. Immersed strength versus lime content relationships after 7 and 28 day curing for montmorillonite clay soils AR-2, AR-3, and AR-7, showing relative effects of Standard and Modified AASHO density compaction, and of U. S. Gypsum dolomitic monohydrate "Type N" and calcitic hydrated limes.**

tents above 6%, dolomitic lime gave higher strengths than calcitic lime. At modified density, the illite clay soils did show significant strength improvements with dolomitic lime in relation to calcitic lime, with strength differences ranging from 150 to 200 psi. A comparison (figure 4) of strengths developed by mixtures of soil AR-8 and each of four limes, compacted at standard density, also shows that dolomitic lime gives higher strengths than calcitic lime, particularly after 28 days of curing.

Strengths at modified density were 150 to 250 psi higher than at standard density for all mixtures except soil AR-4 and calcitic lime (figure 3). Mixtures of soil AR-4 and calcitic lime at modified density tended to slake during immersion.

All strength versus lime content curves (figure 3), except for soil AR-9 and dolomitic lime, show slight or negative slopes above 6% lime, signifying that the optimum lime content of illite clay soils may be at or below 6%. Soil AR-9 with dolomitic lime had an optimum lime content of 8%, the higher lime requirement for maximum strength could be expected since of the three illite clay soils used, soil AR-9 contained by far the highest percentage of clay size material (table II).

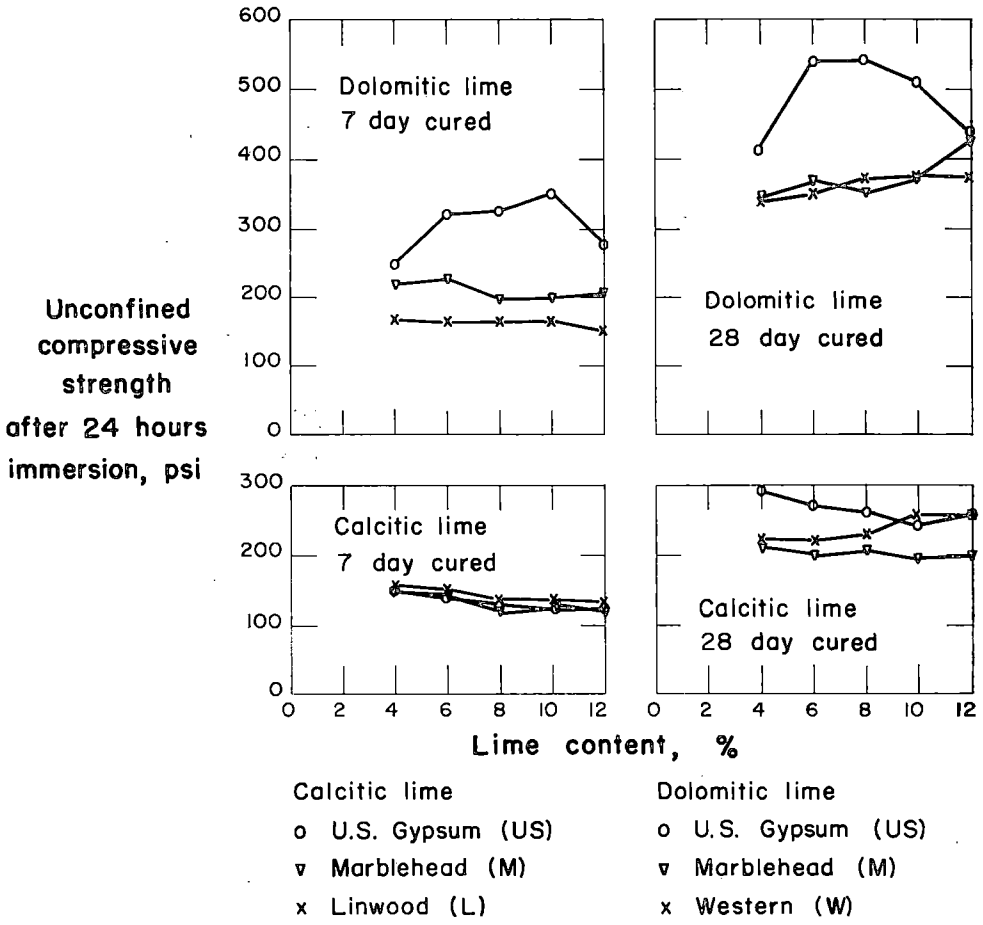


**Fig. 2 Immersed strength versus lime content relationships after 7 and 28 day curing for montmorillonite clay soil AR-2 and Standard AASHO density compaction, showing relative effects of three dolomitic monohydrate "type N" limes and three calcitic hydrated limes.**

**Kaolinite clay soils.**

At both standard and modified density, the maximum strengths obtained with kaolinite clay soils AR-5 and AR-10 were at most only 130 psi higher with dolomitic lime than with calcitic lime (figure 5), and at lime contents of 6 to 8%, both U. S. Gypsum limes gave about equal strengths. With soil AR-6, U. S. Gypsum calcitic lime produced somewhat higher strengths than U. S. Gypsum dolomitic lime, especially in mixtures cured 28 days (figure 5). However, the other calcitic and dolomitic limes (figure 6) gave about the same strengths in mixtures with soil AR-6.

Strengths of all mixtures were significantly improved, 100 to 300 psi,

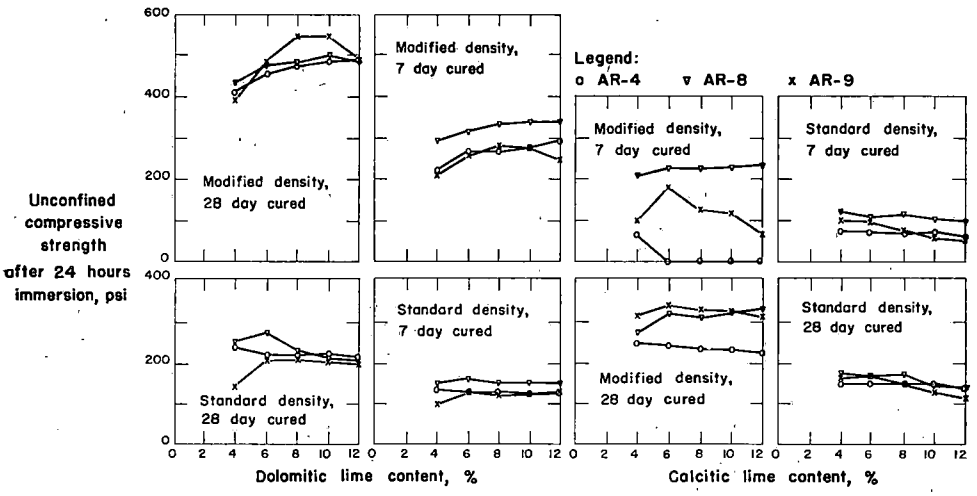


**Fig. 3. Immersed strength versus lime content relationships after 7 and 28 day curing for illite clay soils AR-4, AR-8, and AR-9, showing relative effects of Standard and Modified AASHO density compaction, and of U. S. Gypsum dolomitic monohydrate "Type N" and calcitic hydrated limes.**

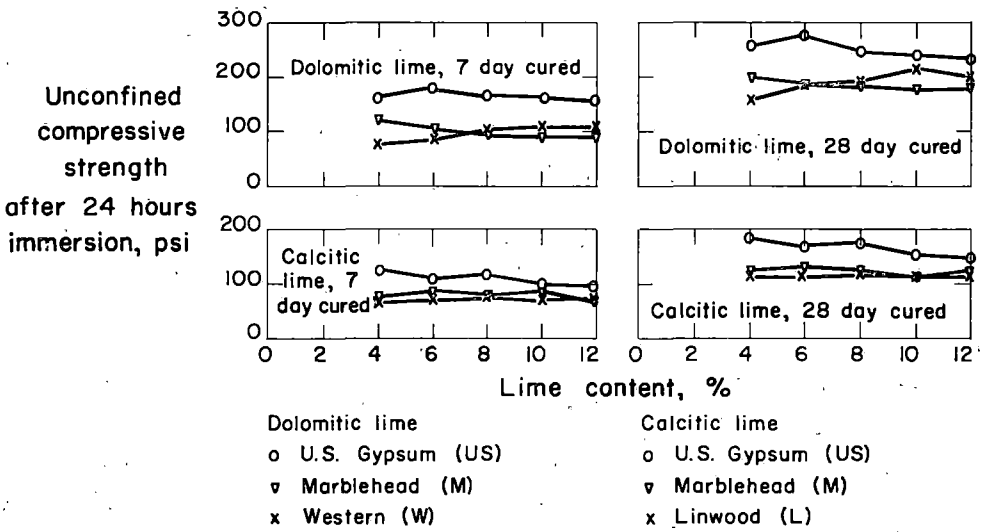


**Fig. 4. Immersed strength versus lime content relationships after 7 and 28 day curing for illite clay soil AR-8 and Standard AASHO density compaction, showing relative effects of three dolomitic monohydrate "Type N" limes and three calcitic hydrated limes.**

by the use of modified density compaction instead of standard.

In general the optimum lime content for the soils was greater with dolomitic lime than with calcitic lime.

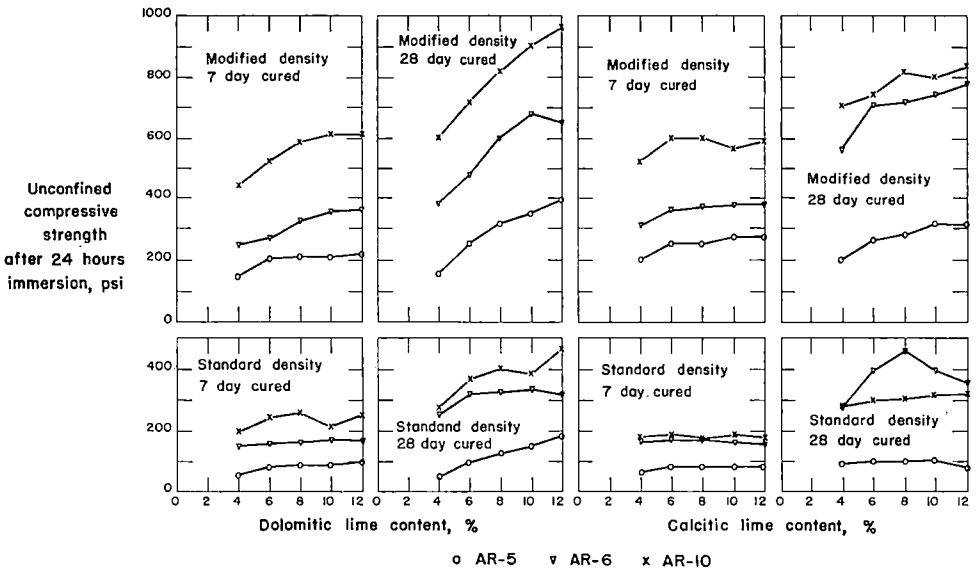


**Fig. 5. Immersed strength versus lime content relationships after 7 and 28 day curing for kaolinite clay soils AR-5, AR-6, and AR-10, showing relative effects of Standard and Modified AASHO density compaction, and U. S. Gypsum dolomitic monohydrate "Type N" and calcitic hydrated limes.**

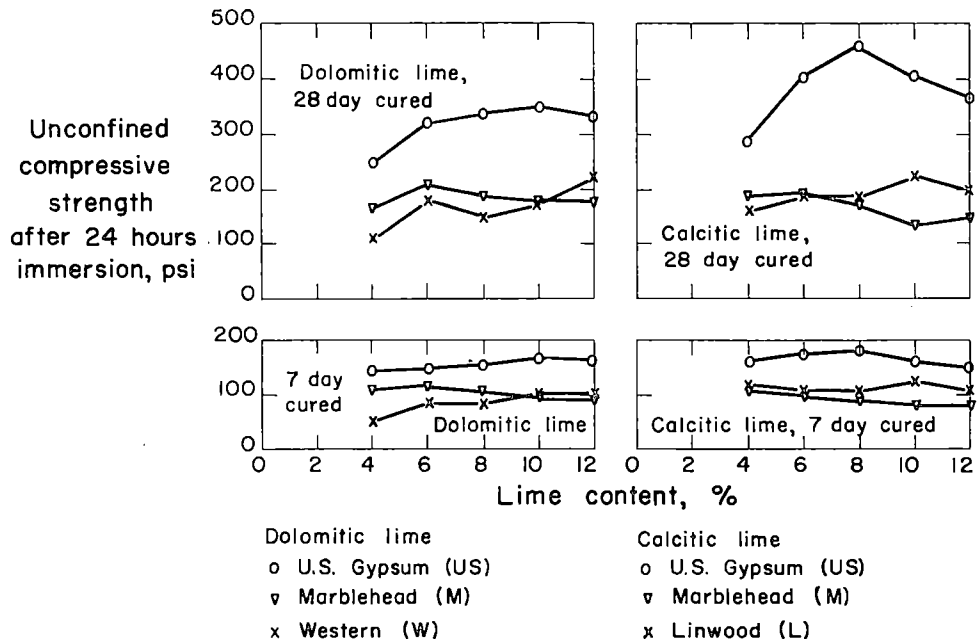


**Fig. 6. Immersed strength versus lime content relationships after 7 and 28 day curing for kaolinite clay soil AR-6 and Standard AASHO density compaction, showing relative effects of three dolomitic monohydrate "Type N" limes and three calcitic hydrated limes.**

## CONCLUSIONS

1. In mixtures with the montmorillonite and illite clay soils dolomitic monohydrate "Type N" lime produces higher immersed strengths than calcitic hydrated lime; more specifically:

   (a) The montmorillonite clay soils show strengths 130 to 250 psi higher at near standard AASHO density and 150 to 300 psi higher at near modified AASHO density, and

   (b) The illite clay soils show strengths 40 to 90 psi higher at near standard AASHO density and 150 to 200 psi higher at near modified AASHO density.

2. Neither dolomitic monohydrate "Type N" lime nor calcitic hydrated lime consistently produce the highest strengths in kaolinite clay soil and lime mixtures. Dolomitic lime produces the highest strengths in two of the three soils tested, and calcitic lime produces the highest strengths in the third.

3. With all soil and lime mixtures studied, modified AASHO density compaction gives immersed strengths 100 to 350 psi higher than standard AASHO density compaction; except for soil AR-2, strengths over 500 psi could be obtained only by use of modified AASHO density compaction.

4. Optimum lime contents for maximum immersed strengths are generally higher when using dolomitic monohydrate "Type N" lime than when using calcitic hydrated lime.

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# LIME AND SODIUM SILICATE STABILIZATION OF MONTMORILLONITE CLAY SOIL

by

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(Highway Research Board Proceedings. 1961.)

## ABSTRACT

The results and interpretations of a laboratory soil stabilization investigation of combination lime and sodium silicate treatments of a montmorillonite rich clay soil are presented. Five kinds of powdered sodium silicate were evaluated in mixtures with the soil and a calcitic hydrated lime. Mixtures with sodium sesquisilicate pentahydrate appeared least affected by elapsed time between mixing and compaction. Hence this sodium silicate was selected for studies of the effect of lime and sodium silicate mix proportions on immersed strength versus curing time and on freeze-thaw resistance.

## INTRODUCTION

This report covers an investigation of the stabilization of a clayey soil with lime and sodium silicate.

Sodium silicate and calcium chloride have long been combined as a grout to seal foundations and tunnels, but the use of this mixture in stabilizing base and subbase courses of pavements has not been very successful. However, combinations of lime and sodium silicate have given satisfactory laboratory results. A prior investigation suggested that the soil, lime, and sodium silicate reaction was such that the final strength of the stabilized soil depended on the elapsed time between the mixing of the soil, lime, sodium silicate, and water combination and the final compaction of the mixture[6]. In field construction of base and subbase courses, several hours may elapse between the final mixing of the soil and the stabilizing agents and completion of compaction. It is of course desirable for this delay to have little or no effect on the strength of the stabilized soil.

The investigation in this report was conducted to determine the following concerning the use of lime and sodium silicate for stabilizing a clayey soil for use as a base or subbase course of a flexible pavement:

1. The effect of elapsed time between mixing and molding on the strength and dry density of the soil treated with calcitic hydrated lime and various sodium silicates.
2. The seven day cured strengths of the soil treated with varying percentages of calcitic hydrated lime and the sodium silicate whose strength is least affected by elapsed time.
3. The effect of curing time on the strength of the stabilized soil.
4. The resistance of the stabilized soil to freeze-thaw action.

**Materials**

Sodium silicate is a chemical compound manufactured by melting soda ash and selected silica sand ($SiO_2$) together at high temperatures. The resulting product is a water soluble glass. This glass is ground into various sized particles for different uses. (Fifteen different solid sodium silicates are manufactured by one company alone.) These sodium silicates vary in alkali to silica ratio, water content and particle size, depending on their proposed use. Sodium silicates are used as detergents, deflocculating agents, films and coatings, sizing, corrosion control agents, bleaching agents, adhesives, and cements[13].

Lime is strictly defined as CaO, but the term is commonly used to include quicklime and hydrated lime. These are oxides and hydroxides of calcium and calcium magnesium. Commercial lime is manufactured by heating a crushed carbonate rock such as limestone, $CaCO_3$, causing a release of $CO_2$ and leaving a calcitic (high calcium) lime or CaO. Another type of carbonate rock called dolomite is also used for lime manufacture. Since dolomitic rocks have various ratios of $CaCO_3$ and $MgCO_3$, the resulting lime, called dolomitic lime, is a mixture of CaO and MgO. Quicklime is another term for lime composed of calcium or calcium and magnesium oxides. Quicklime reacts readily with water to produce slaked lime or hydrated lime, in which all the CaO is converted to $Ca(OH)_2$. The MgO of dolomitic quicklime hydrates less rapidly, and in normal hydration processes most of the magnesium remains as MgO[11].

**Previous Investigations**

Sodium silicate has been used to improve building materials for over a century. A pioneer in the commercial development of soluble silicates, proposed in the early Nineteenth Century that soluble silicates could be used as a hardening agent for artificial stone. He also recognized the reaction of silicates with calcium compounds. During the Nineteenth Century several patents were issued for the manufacture of artificial stones from solutions of silicates mixed with solutions of calcium compounds[20].

As early as 1910, sodium silicate is mentioned as being used as a dust palliative. The application of a solution of sodium silicate followed by an

application of a soluble aluminum or calcium salt causes the precipitation of an aluminum or calcium silicate in place of the road stone[10]. This led to the development of macadam roads in which the crushed stone was held in place by a cementitious matrix formed by a slurry of finely divided limestone and sodium silicate[9, 15, 20].

Around 1915 the effectiveness of cement grout was found to be increased by preceeding the grout with an injection of alum followed by sodium silicate. Apparently the resulting slippery gel coats the granular surfaces and causes greater penetration of the cement grout[20]. Later, sodium silicate was injected as a grout into deep foundations followed by an injection of calcium chloride[16]. The combination of these two chemicals forms an insoluble gel which fills the voids and prevents seepage of water or the continuance of any settlement[20]. This injection method with modifications has been extensively utilized in foundation work and in controlling seepage, but the method is not practical in solidifying the soil within three feet of the surface[17]. Another injection method, in which the precipitation reaction is much slower, utilizes a solution containing sodium silicate and sodium bicarbonate. The gel formed is not the insoluble calcium silicate and gradually deteriorates, nor does it have the strength of the calcium silicate gel[14].

Sodium silicate alone was investigated as a stabilizer for various soils. The conclusion was that, while sodium silicates showed promise as stabilizing agents, the soil should be studied and evaluated prior to treatment, since some soils gave a negative response to the treatment[12].

Investigations of soil stabilization at Iowa State University indicate that combinations of sodium silicate and lime show promise, because the reaction is less rapid than that between sodium silicate and calcium chloride. The chemical reaction produces sodium hydroxide which has a desirable effect on soil stability. These incomplete investigations indicate that a wide range of soils from fine sands to montmorillonitic clay soils can be stabilized with lime and sodium silicate[7].

**Mechanism of Sodium Silicate Stabilization**

The exact mechanism of sodium silicate soil stabilization is not known. Sodium silicate was found to react with the clay minerals present in the soil, but at detailed study was not made of the nature or possible mechanism of the assumed reaction[12].

Sodium silicate is used in soil stabilization mainly because it reacts with soluble calcium salts in water solutions to form insoluble, gelatinous calcium silicates[9]. This reaction is advantageous, since water is necessary for the proper compaction of a soil to a desired density[9].

That hydrated calcium silicates are cementing agents has been fairly well established[5]. Hydration, a process of a solid combining with water[8], is the essential mechanism of setting or hardening of a gel[19]. The exact

nature of the bonding action of hydrated calcium silicates has been the subject of much investigation, but it has not been fully explained. In addition to its cementing properties, the gelatinous calcium silicate improves the stability of the soil by filling voids, thereby excluding water from the soil. Excessive moisture in ordinary soil causes loss of strength and reduces the soil stability[9].

## MATERIALS AND METHODS

### Materials Used

*Properties of soil.* Kansan till, comprised of approximately equal portions of sand, silt and claysized materials, was the soil used throughout the

TABLE I. SAMPLING LOCATION OF THE SOIL

| | |
|---|---|
| Location: | SW¼SE¼ of Section 23, Tier 68 North, Range 31 West, Benton Township, Ringgold County, Iowa |
| Geological description: | Kansan-age glacial till, plastic, calcareous |
| Soil series: | Shelby |
| Horizon: | C |
| Sampling depth: | 4½ - 10½ feet |
| Laboratory No. | 409-12C |

TABLE II. PROPERTIES OF THE SOIL

| | |
|---|---|
| Physical Properties: | |
| Textural composition percent by weight: | |
| Gravel (above 2.0 mm) | 0 |
| Sand (2.0 - 0.074 mm) | 33.9 |
| Silt (0.074 - 0.005 mm) | 27.6 |
| Clay (below 0.005 mm) | 39.5 |
| Colloidal (below 0.001 mm) | 29.0 |
| Textural classification:* | Clay |
| Atterberg limits: | |
| Liquid limit, % | 39.4 |
| Plastic limit, % | 16.5 |
| Plasticity index, % | 22.9 |
| Shrinkage limit, % | 8.2 |
| Engineering classification† | A-7-6(11) |
| Predominant clay mineral:‡ | Montmorillonite |
| Chemical Properties: | |
| Cation exchange capacity (whole soil) m.e./100 gm | 29.46 |
| pH | 8.0 |
| Carbonates, % | 6.4 |
| Organic matter, % | 0.2 |

*Textural classification was by the triangular chart developed by the U. S. Bureau of Public Roads (18, p. 48): 0.074 mm was used as the lower limit of sand fraction.

†AASHO Method M145-49 (1).

‡Determined by X-ray analysis of the fraction passing the No. 200 sieve.

investigation. This soil was chosen because it is considered an in-between soil; it is not the worst soil found in Iowa for road building, and it is not the best. Another reason for this choice was some data on Kansan till had been collected in an investigation of lime-sodium silicate stabilization[6]. The field location of the sample is given in table I, and physical and chemical properties are given in Table II.

*Properties of lime.* Calcitic hydrated lime was the only lime used in the investigation, because calcitic hydrated lime gave better results than other type limes[6].

The properties of the commercial grade lime used are shown in table III. The lime was produced by a plant of the U. S. Gypsum Company, Chicago, Ill.

TABLE III. PROPERTIES OF THE LIME

| | |
|---|---|
| Chemical analysis, percent by weight: | |
| Silicon dioxide | 0.28 |
| Iron and aluminum oxide | 0.6 |
| Magnesium oxide | 0.59 |
| Sulfur trioxide | 0.25 |
| Carbon dioxide | N.D.* |
| Total calcium oxide | 73.82 |
| Available calcium oxide | 70.3 |
| Loss on ignition | 24.1 |
| Combined $H_2O$ | N.D.* |
| Processing location: | New Braunfels, Texas |
| Trade name: | Kemikal, Hydrated |

*Not determined.

*Properties of sodium silicates.* Five powdered sodium silicates were used. These sodium silicates varied in molecular ratio of $Na_2$ to $SiO_2$ and in degree of hydration. By using sodium silicates that varied in these properties it was hoped that some indication of the influence of these variables on the final results would be obtained. Powdered sodium silicates were used because adding the sodium silicate in a liquid form causes the soil, lime, and sodium silicate mixture to set up or solidify too rapidly to permit proper molding of specimens[6].

The sodium metasilicate enneahydrate was an analytical grade reagent, catalog No. 2290, General Chemical Division, Allied Chemical and Dye Corporation. The other four sodium silicates, sodium metasilicate pentahydrate, sodium metasilicate anhydrous, sodium sesquisilicate pentahydrate and sodium orthosilicate, were commercial grade sodium silicates (table IV). The sodium silicates will be identified by their laboratory designations.

**Method of Procedure**

*Preparation of mixtures.* The required amount of air dried soil passing the No. 10 sieve was dry mixed with the powdered lime and sodium silicate

TABLE IV. PROPERTIES OF THE SODIUM SILICATES

| Laboratory designation | Trade name | Chemical name and formula | Molecular ratio $Na_2O:SiO_2$ | $H_2O$ % | $Na_2O$ % | $SiO_2$ % |
|---|---|---|---|---|---|---|
| Silicate A | Reagent | Sodium metasilicate enneahydrate $Na_2SiO_3 \cdot 9H_2O$ | 1:1 | 57.1 | 21.8 | 21.1 |
| Silicate B* | Metso Anhydrous | Sodium metasilicate anhydrous $Na_2SiO_3$ | 1:1 | 0.5 | 51.0 | 45.5 |
| Silicate C* | Metso Granular | Sodium metasilicate pentahydrate $Na_2SiO_3 \cdot 5H_2O$ | 1:1 | 41.7 | 29.5 | 28.7 |
| Silicate D* | Metso 99 | Sodium sesquisilicate pentahydrate $Na_3HSiO_4 \cdot 5H_2O$ | 3:2 | 38.1 | 36.7 | 24.2 |
| Silicate E* | Metso 200 | Sodium orthosilicate concentrated $Na_4SiO_4$ | 2:1 | 9.5 | 60.8 | 27.5 |

*Data from Philadelphia Quartz Company, Philadelphia, Pennsylvania.

additive for two minutes by a Hobart Model C-100 mixer modified by attaching a scraper blade to prevent the mixture from caking in the mixing bowl. Sufficient carbon dioxide-free distilled water was added to bring the mixture to the desired moisture content, and mixing was continued for two minutes. The mixture was then hand-mixed long enough to loosen any of the material stuck to the sides of the mixing container. Immediately after mixing, the mixture was placed in a covered container and allowed to age.

This order of mixing was chosen for the following reasons. In the field the probable method of mixing would be either to mix the lime with the soil, add the sodium silicate and remix, then add needed water and mix again; or to mix the lime and sodium silicate with the soil and then add the water and remix. Because the latter method seemed to be the simplest and most likely to be employed in the field, it was chosen for this investigation. The water was not added until the lime and sodium silicate were thoroughly mixed with the soil so that the best possible distribution of reaction products would be obtained. Carbon dioxide-free distilled water was used to reduce the number of variables involved in the investigation. The mixture was aged in a covered container to prevent evaporation of moisture and to prevent the lime from reacting with the carbon dioxide in the air.

All additive percentages used in this study are based on the weight of oven dry soil. Moisture contents are based on the weight of the oven dry

treated soil.

*Molding of specimens.* After being aged the mixture was molded into cylindrical shaped specimens, two inches high and two inches in diameter, with a density near AASHO density (AASHO Method T99-57) (2)[4].

*Curing of specimens.* Specimens made for the strength vs. ageing time study, and for the best lime sodium silicate mixture study were cured for 7 days in a moist curing room. The relative humidity in the room was 95% or higher; the temperature was maintained at 75° F ± 5° F. After being molded and before being placed in the curing room, each specimen was sealed in wax paper to reduce evaporation of moisture and to prevent carbon dioxide of the air from reacting with the lime during curing. Specimens for the strength vs. curing time study were cured for 3, 7 and 28 days.

*Strength testing.* All specimens except those specifically designated were immersed in distilled water for twenty-four hours after curing and before being tested for strength. The immersion reduces or destroys any apparent cohesion in the soil and tests the true cohesion and other stabilizing effects of the soil, lime, and sodium silicate mixture. Strengths were measured by unconfined compression testing. The rate of loading was 0.1 inches per minute. The maximum load causing failure was considered the compressive strength of the specimen. All of the unconfined compressive strengths were maintained within 10% of the average values as required by ASTM Designation: C109-54T[3], or tests were repeated.

*Freeze-thaw testing.* A modified British freeze-thaw test was used to evaluate the resistance of selected mixtures to the seasonal influence of freezing and thawing. Five specimens from each mixture were molded and cured for 7 days, then immersed in distilled water at room temperature for twenty-four hours. Three specimens from each mixture were then tested for unconfined compressive strength; one specimen was left immersed for fourteen more days; and the last specimen was exposed alternately to temperatures of −5° C and 25° C for fourteen cycles, each cycle lasting twenty-four hours. After these fourteen day treatments the freeze-thaw specimen and the immersed specimen were tested for unconfined compressive strength[4]. A comparison of these values was used to determine the ability of the lime and sodium silicate stabilized soil to resist freezing and thawing.

## PRESENTATION AND DISCUSSION OF TEST RESULTS

### Effect of ageing time on dry density and strength

This phase of the investigation had two purposes. The first was to determine the effect of ageing time, defined as the elapsed time between mixing and molding, on the strength and dry density of specimens of soil treated with calcitic hydrated lime and each of the sodium silicates. The second was to determine which sodium silicate gave the highest strength and was least

affected by ageing time.

The number of variables affecting the results were reduced to a minimum. Previous laboratory work with lime and sodium silicate soil stabilization indicated that the elapsed time between the addition of water to the soil, lime, and sodium silicate mixture and the completion of molding greatly affected the density of the specimen, and suggested that strength might likewise be affected. It was also found that the lime and sodium silicate combination that gave the best results in stabilizing Kansan till was 6% calcitic hydrated lime and 6% sodium metasilicate pentahydrate[6]. Since the aqueous portion of the sodium silicate does not react with the lime, although it does affect the reaction, it was decided to compare the effectiveness of the sodium silicates on an equivalent anhydrous weight basis. Therefore, combinations of 6% lime and an amount of each sodium silicate, such that the anhydrous portion was equal in weight to the anhydrous portion of 6% sodium metasilicate pentahydrate, were used in the study.

The ageing time was the time, recorded to the nearest minute, between the addition of water to the dry soil, lime, and sodium silicate mixture and the final compaction of the specimen. Specimens with ageing times of approximately 9 minutes and 1, 2, 3, and 5 hours were molded. The specimens were weighed and measured immediately after molding. At the same time samples were taken from the mixture to determine the molding moisture content of the specimen. This molding moisture content includes the water present in the sodium silicate as well as the water added. The dry density of the specimen at the time of molding was calculated from the equation:

$$\text{Dry density} = \frac{1.2125 \times \text{weight of specimen}}{(1 + \text{moisture content}) \times \text{height of specimen}}$$

To determine the influence of molding moisture content on the effect of ageing time on strength and dry density, the soil, lime, and sodium silicate mixtures were molded at various moisture contents. The optimum moisture content for near 100% standard AASHO dry density of Kansan till stabilized with 6% calcitic hydrated lime was found to be 17.0%. For the soil, lime, and sodium silicate mixtures with each of the sodium silicates, it was originally planned to use five different molding moisture contents; two below this optimum for the soil and lime mixture and three above this optimum. These planned moisture contents were 13, 15, 17, 19 and 21%. The amount of water needed to reach these molding moisture contents was calculated; however, because the chemical reaction between the lime and sodium silicate differed for each sodium silicate, the actual molding moisture contents differed from the planned molding moisture contents by varying amounts.

The specimens after being molded were moist cured for 7 days and then were immersed in distilled water for 24 hours before they were tested

for ultimate unconfined compressive strength. Unless otherwise stated, the term *strength* when used in this report means unconfined compressive strength after 24 hours immersion.

## Discussion

Specimens of Kansan till stabilized with 6% calcitic hydrated lime and varying amounts of sodium silicates were molded. The anhydrous portion of 6% sodium metasilicate pentahydrate used was equal in weight to the anhydrous portion of each of the other silicates used. This was done as a control. Different molding moisture contents change the effect of ageing on dry densities (figure 1). The percentages of the different sodium silicates are based on the weight of the sodium silicate in its normal state of hydration. The points are the average of three specimens, molded at as nearly the same time as possible, plotted at the average ageing time of the specimens. The molding moisture content shown is the range of the molding moisture contents of the specimens molded at the five different ageing times.

In all except one of the mixtures of soil, lime, sodium silicate, and water the highest dry densities were obtained at ageing times near zero (figure 1).

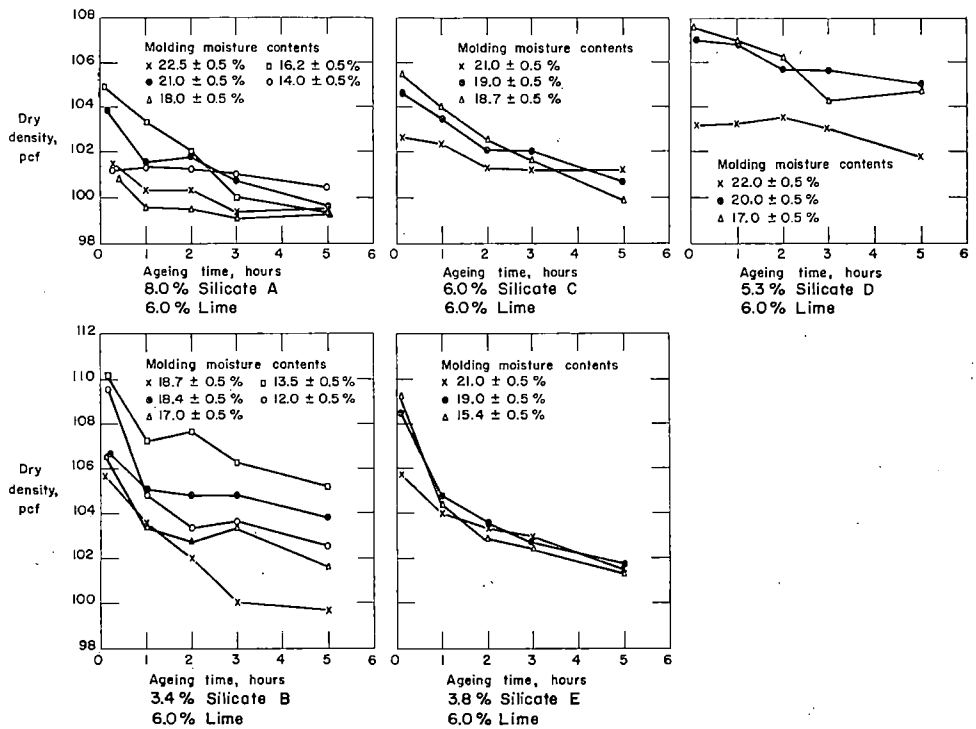


**Fig. 1. Influence of molding moisture content on the effect of ageing time on dry density at time of molding Kansan till stabilized with 6% calcitic hydrated lime, and indicated percentages of various sodium silicates.**

This one mixture showed a higher dry density after 2 hours of ageing than at shorter ageing times; however, this density was so close to the dry densities of the same mixture with greater and lesser ageing times that it seems safe to consider that the discrepancy is due to experimental error. As the ageing time increased, the dry density of the specimens molded from all the mixtures decreased, thus confirming the indications of the previous Iowa State work[6].

The molding moisture content influenced dry density, but the molding moisture content that gives highest dry density at the shortest ageing time does not usually give highest dry density at the longest ageing time. This indicates that the optimum moisture content for maximum dry density varies with ageing period.

An explanation of the dependence of dry density and optimum moisture content for maximum dry density on ageing time is related to the chemical reaction between lime and sodium silicate. As water comes in contact with lime and sodium silicate, the chemicals dissolve and then react to form an insoluble calcium silicate gel. Since lime is slowly soluble, the quantity of gel formed is gradually increased as the reaction between the lime and sodium silicate continues. The gel absorbs water and at the same time begins to hydrate. As the gel hydrates, it hardens. The longer this process continues, the more gel is converted into a hydrated calcium silicate.

Both the processes of gel formation and hydration remove free water from the mixture. This free water is necessary to act as a lubricant for the compaction process[9]. The gel may possibly act as a lubricant in place of the absorbed water, but further investigation is necessary to determine this. The change in the amount of lubricant present may account for the change in optimum moisture content for maximum dry density.

As the gel hydrates, the character of the soil changes because the hydrated gel is cementing soil particles together, thus changing the effective gradation of the mixture and increasing its resistance to the compaction force. This not only affects the optimum moisture content for maximum dry density but it also affects the maximum dry density itself. Since the amount of compactive effort applied in the standard density test is held constant, an increase in soil resistance to compaction will cause a decrease in density. The foregoing seems to be an explanation for the dependence of dry density and optimum moisture content for maximum dry density on ageing time.

The type of sodium silicate also influenced dry density and the amount of decrease in dry density with ageing time. Highest dry densities were obtained with silicates B and E. But mixtures with these silicates also had the greatest decrease in dry density with increase in ageing time (figure 1). Silicates A and C gave the lowest dry densities, and they had a smaller decrease in dry density with increase in ageing time. The dry densities achieved by silicate D mixtures were between those of the other two groups, and the decrease in dry density with ageing time appeared to be smallest.

The explanation of the above possibly is related to the gel forming ability of the different sodium silicates. In the explanation given earlier for the dependence of dry density on ageing time, emphasis was placed on the controlling function of the amount of gel present. A difference in the rate of gel formation by different sodium silicates would produce different rates of decrease of dry density with ageing time. A difference in the rate of gel formation might also account for the difference in dry densities obtained from the different sodium silicate mixtures.

**Strength**

As was true with dry densities, different molding moisture contents change the effect of ageing on strength (figure 2). The percentages of the different sodium silicates are based on the weight of the sodium silicate in its normal state of hydration. The points are the average of three specimens, molded at as nearly the same time as possible, plotted at the average ageing time of the specimens. The molding moisture contents shown are the range of the molding moisture contents of the speciments molded at the five different molding times.

It is difficult to make any general statements about the effect of ageing time on strength that would apply to all of the mixtures of soil, lime, and sodium silicate. Each mixture was affected differently by water content and ageing time (figure 2).

Mixtures made with silicate A showed a decrease in strength with an increase in ageing time. The mixtures with the highest and lowest molding moisture contents had strengths that were least affected by ageing time. At an ageing time of 5 hours, the silicate A mixture with the highest molding moisture content had the highest strength.

Silicate B mixtures showed a more marked decrease of strength with ageing time than silicate A mixtures, but the strengths of the silicate B mixtures in general were higher than those of the silicate A mixtures molded at approximately the same moisture content. Silicate B mixtures could not be molded at moisture contents above 19.5% because the material solidified in the mixer.

Based on the results obtained with silicate A and silicate B, it was decided to evaluate the other sodium silicate mixtures at only the three highest moisture contents.

Silicate C mixtures also decreased in strength as ageing time increased. Again the mixture with the highest molding moisture content showed the least effect of ageing time on strength and had the highest strength with an ageing time of 5 hours.

The molding moisture content had a strange effect on the strength of the silicate D mixtures. At the lowest molding moisture content the silicate D mixture behaved the same as the aforementioned silicate mixtures, but at the next higher molding moisture content the strength of the mixture

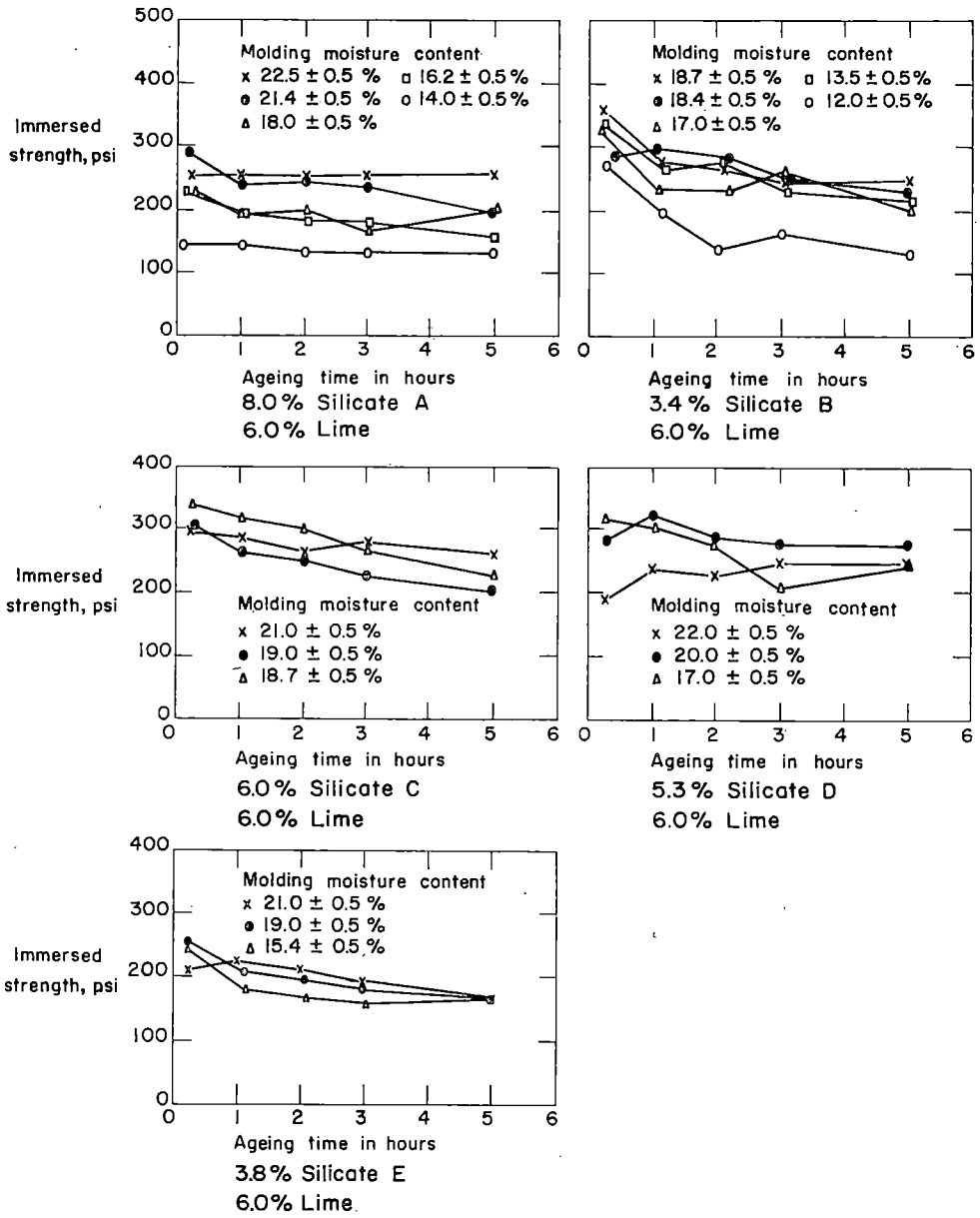


**Fig. 2. Influence of molding moisture content on the effect of ageing time on 7 day cured, 1 day immersed unconfined compressive strength; Kansan till stabilized with 6% calcitic hydrated lime, and indicated percentages of various sodium silicates.**

increased between ageing times of 0 and 1 hour and then decreased until at an ageing time of 5 hours the strength was approximately the same as

at an ageing time of 0.25 hours. At the highest molding moisture content the mixture actually showed a gain in strength with an increase in ageing time. However, the highest strength at an ageing time of 5 hours was with the silicate D mixture molded at the middle moisture content.

Silicate E mixture molded at the highest molding moisture content displayed the same characteristic as the silicate D mixture molded at the middle molding moisture content. The other two molding moisture contents caused the strength of the mixture to decrease as the ageing time increased. It is noteworthy that, at an ageing time of 5 hours, the strength appears to be independent of molding moisture content.

The optimum moisture content for maximum strength changes with ageing time and is not the same as the optimum moisture content for maximum dry density (figures 1, 2). Strength is not correlated with dry density, which means that it is not possible to achieve a desired strength in the field by compacting the treated soil to a specified dry density. Nor is it possible to check field strengths by checking dry densities.

An explanation of the decrease in strength with increase in ageing time may be given along the same line as the explanation for the decrease in dry density. If the amount of gel formed by the reaction between the lime and the sodium silicate increases with time, then as the ageing time increases the amount of gel present at the time of molding increases. In the absence of any information to the contrary, it is assumed that the gel forming ability of the mixture is unaffected by the compaction. The structure of this gel can be broken by compaction and will reform, but the reformed gel will not form as strong a cementing agent[19]. The amount of gel present at a given time depends on the amount of water in the mixture and on the rate of gel formation. While gel formation rates of lime and sodium silicate mixtures have been studied, no investigation has been made to determine the rate of this gel formation in the soil, lime, sodium silicate and water mixture. From the results of this investigation it has not been possible to determine the exact effect of the $Na_2O$:SiO ratio and the degree of hydration of the sodium silicate on the strength of the lime and sodium silicate stabilized soil, but it is probable that these properties affect the rate of gel formation. This is only a possible explanation for the observed decrease of strength with increase of ageing time, but it does account for some of the results of this investigation.

*Selection of sodium silicate to be further evaluated.* To assist in determining the sodium silicate whose strength was least affected by the ageing time, the following criterion was adopted. The lime and sodium silicate stabilized soil should have a strength of over 225 psi for the specimens molded after an ageing time of 5 hours. This criterion recognizes that a stabilized soil must be frost resistant. Previous work had shown that materials with a seven day cured, one day immersed unconfined compressive strength between 200 to 250 psi probably will satisfactorily resist freeze-thaw action[4].

Further, the criterion recognizes that field compaction may not be completed before 5 hours after the water has been added to the lime and sodium silicate treated soil. Therefore any mixture that does not have the minimum strength at an ageing time of 5 hours should be eliminated from further consideration.

Table V lists the lime and sodium silicate mixture with each sodium silicate that either best satisfies the above criterion or comes closest to satisfying it. From this table it is seen that silicate D mixture not only satisfies the criterion but also has the least overall decrease in strength between ageing times of approximately 0.25 hours and 5 hours. Therefore silicate D was chosen as the admixture for the remaining phases of the investigation.

Because the strengths of silicate D mixtures showed such a slight dependence on ageing time, it was decided to allow the mixtures used in the remainder of the investigation to age two hours. This time of 2 hours was chosen because the elapsed time between mixing and compaction in the field, using a multi-pass mix-in-place procedure to mix the stabilized soil, is from 2 to 3 hours[21].

## DETERMINATION OF OPTIMUM COMBINATION OF LIME AND SILICATE D

The purpose of this study was to determine the combination of lime and silicate D that produced the best results in stabilizing Kansan till, and also to find the optimum molding moisture content for maximum strength of this combination.

Sufficient soil for three specimens was mixed with the desired amount of water to obtain a moisture content on the dry side of optimum moisture content for maximum dry density. The mixture was allowed to age 2 hours and then specimens were molded, measured and weighed[4]. Two samples of the mixture were taken for moisture content determination at the time of molding. The procedure was repeated three times with the moisture content of the mixture increased approximately 2% for each repetition.

Specimens were molded with 0, 2, 4, and 6% calcitic hydrated lime plus 0, 2, 4, and 6% silicate D. The specimens molded with varying moisture content were cured for 7 days, immersed in distilled water for 24 hours, and then tested for unconfined compressive strength.

Average values of dry density (at time of molding) and of immersed strength were calculated for each moisture content. From these data comparisons were made of the optimum moisture requirements for maximum dry density and maximum strength obtained from curves such as those for the 4% silicate D plus 0, 2, 4, and 6% calcitic hydrated lime treated Kansan till mixtures (figure 3).

Table VI is a summary of the results obtained from these curves. In cases typified by the 4% lime and 4% silicate D mixture (figure 3), where

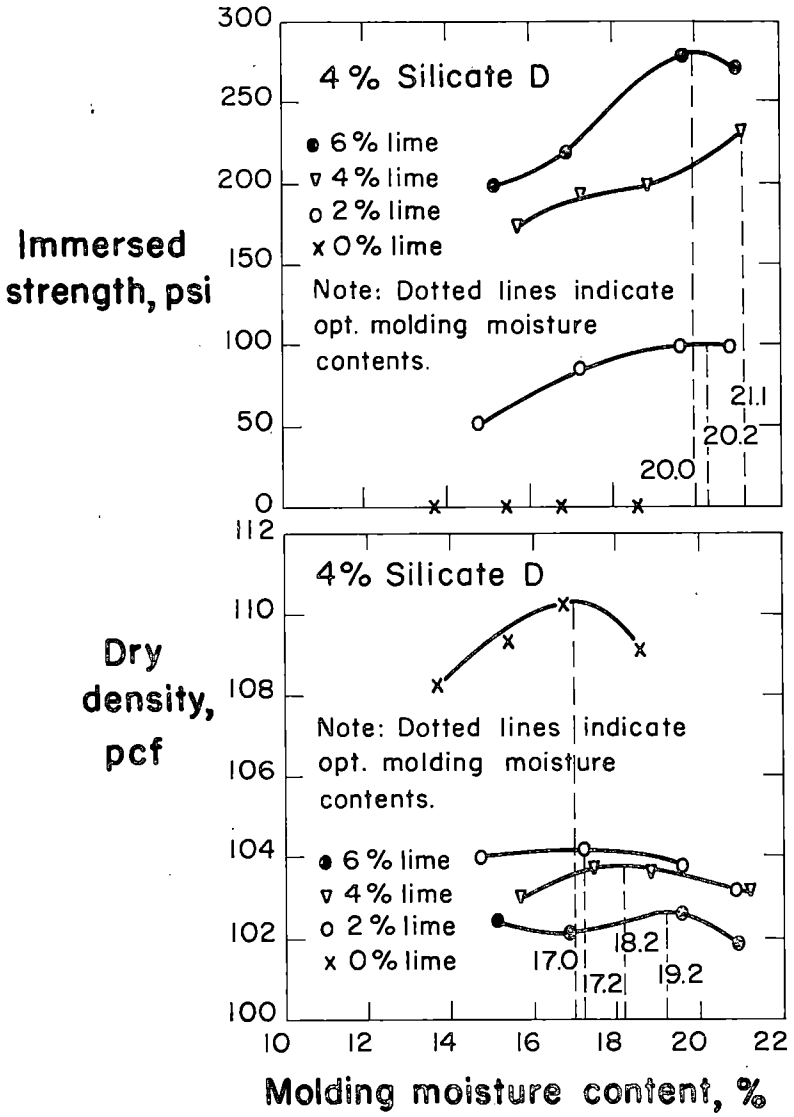


Fig. 3. Typical moisture density and moisture strength curves for calcitic hydrated lime and silicate D soil mixtures studied. (The molding moisture contents indicated for each curve are the selected optimums for that combination of lime and silicate D.)

the moisture strength curve did not pass through a maximum, the highest molding moisture content at which specimens were molded was taken as the optimum moisture content for maximum strength.

To evaluate this data an iso-strength contour chart was used (figure 4).

TABLE V. LIME AND SODIUM SILICATE MIXTURES FOR EACH SODIUM SILICATE THAT BEST SATISFY CRITERION FOR FURTHER EVALUATION

| Sodium silicate content Type** | % | Molding moisture content,* % | Strength At approx. 0.25 hrs,† psi | At approx. 5 hrs,‡ psi | Amount of decrease,§ % |
|---|---|---|---|---|---|
| A | 8.0 | 22.5 ± 0.5 | 251 | 231 | 7.99 |
| B | 3.4 | 18.4 ± 0.5 | 289 | 225 | 22.15 |
| C | 6.0 | 21.0 ± 0.5 | 301 | 240 | 20.25 |
| D | 5.3 | 20.0 ± 0.5 | 277 | 267 | 3.61 |
| E | 3.8 | 21.0 ± 0.5 | 209 | 167 | 20.10 |

*Range of the molding moisture contents of the specimens molded at five different molding times.
†7 day cured, 1 day immersed unconfined compressive strength with approximately 0.25 hours ageing time.
‡7 day cured, 1 day immersed unconfined compressive strength with approximately 5 hours ageing time.
§$\frac{(\text{str @ 0.25 hrs}) - (\text{str @ 5 hrs}) \times 100}{(\text{str @ 0.25 hrs})}$
**Legend for sodium silicate type:
A — Sodium metasilicate enneahydrate
B — Sodium metasilicate anhydrous
C — Sodium metasilicate pentahydrate
D — Sodium sesquisilicate pentahydrate
E — Sodium orthosilicate

TABLE VI. SUMMARY OF TEST RESULTS FOR DETERMINATION OF OPTIMUM COMBINATION OF CALCITIC HYDRATED LIME AND SILICATE D.

| Lime, % | Silicate D, % | Maximum strength,* psi | Maximum dry density,† psi | Optimum molding moisture content, for Maximum strength,* % | Maximum dry density,† % |
|---|---|---|---|---|---|
| 0 | 0 | 0 | 117.3 | ‡ | 14.0 |
| 0 | 2 | 0 | 109.1 | ‡ | 15.6 |
| 0 | 4 | 0 | 110.2 | ‡ | 17.0 |
| 0 | 6 | 0 | 110.7 | ‡ | 18.2 |
| 2 | 0 | 128 | 109.6 | 18.0 | 18.0 |
| 2 | 2 | 120 | ‡ | 19.3 | ‡ |
| 2 | 4 | 100 | 104.1 | 20.2 | 17.2 |
| 2 | 6 | 76 | 105.0 | 16.2 | 16.8 |
| 4 | 0 | 128 | 108.2 | 18.8 | 18.4 |
| 4 | 2 | 205 | ‡ | 19.0 | ‡ |
| 4 | 4 | 232 | 103.8 | 21.1 | 18.2 |
| 4 | 6 | 164 | 104.0 | 20.3 | 19.2 |
| 6 | 0 | 105 | 106.6 | 18.6 | 18.7 |
| 6 | 2 | 242 | ‡ | 20.9 | ‡ |
| 6 | 4 | 285 | 102.6 | 20.0 | 19.2 |
| 6 | 6 | 221 | ‡ | 21.5 | ‡ |

*7 day cured, 1 day immersed unconfined compressive strength.
†Dry density at time of molding.
‡Unable to determine from data.

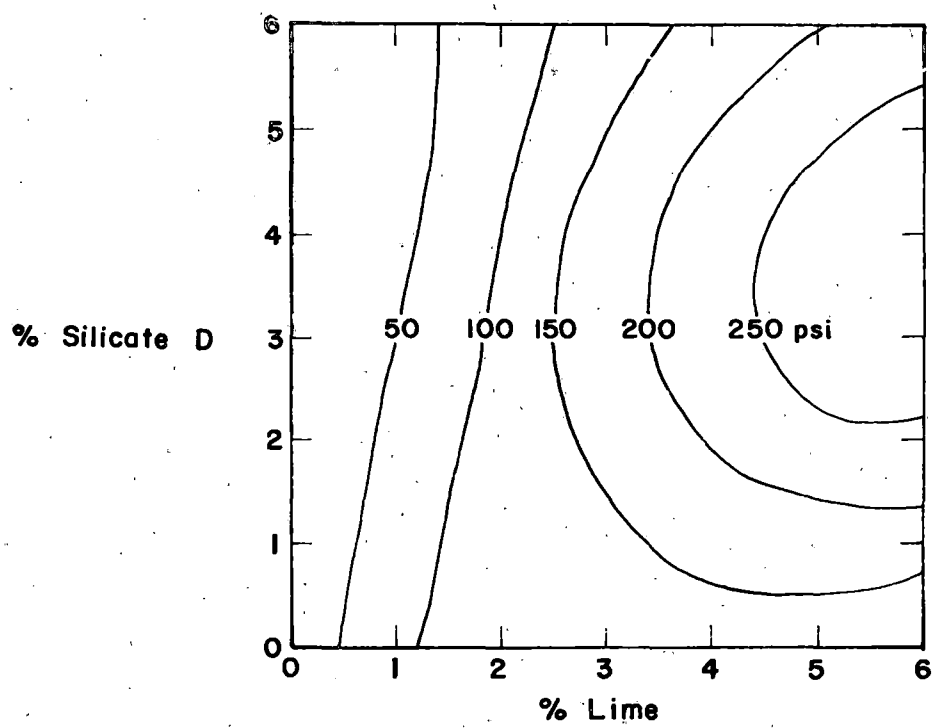


Fig. 4. Iso-strength contour chart showing variation of 7 day cured, 1 day immersed unconfined compressive strength of Kansan till stabilized with calcitic lime and silicate D.

## Discussion

Silicate D used alone produced no increase in the immersed strength of the soil (table VI). The use of amounts of silicate D greater than 2% had no appreciable effect on the maximum dry density. About the only effect silicate D had was to increase the optimum moisture content for maximum dry density. However, when calcitic hydrated lime was also added to the soil, the effect of silicate D on maximum dry density remained the same but the effect on optimum moisture content for maximum dry density was unpredictable.

In the iso-strength contour chart for Kansan till stabilized with calcitic hydrated lime and silicate D, the optimum mix design for maximum strength of the combinations tested was approximately 6% lime and 4% silicate D. For this mixture the optimum molding moisture content for maximum strength is on the wet side of optimum for maximum dry density. That this was not true for all the combinations of lime and silicate D tested (table VI) is further indication that strength is not correlated with dry density in lime and sodium silicate stabilized soil.

### Effect of Curing Time on Strength

Based on the results obtained in the optimum lime silicate D study the following mixtures were selected for an investigation of the effect of length of curing time on one day immersed unconfined compressive strength: 6% lime, 1% silicate D; 6% lime, 2% silicate D; 6% lime, 3% silicate D; and 6% lime, 4% silicate D.

Specimens from each of the above mixtures were molded after allowing the mix to age for two hours, then cured for 3, 7, and 28 days. At the end of each curing period the samples were immersed in distilled water for 24 hours and then tested for unconfined compressive strength (figure 5).

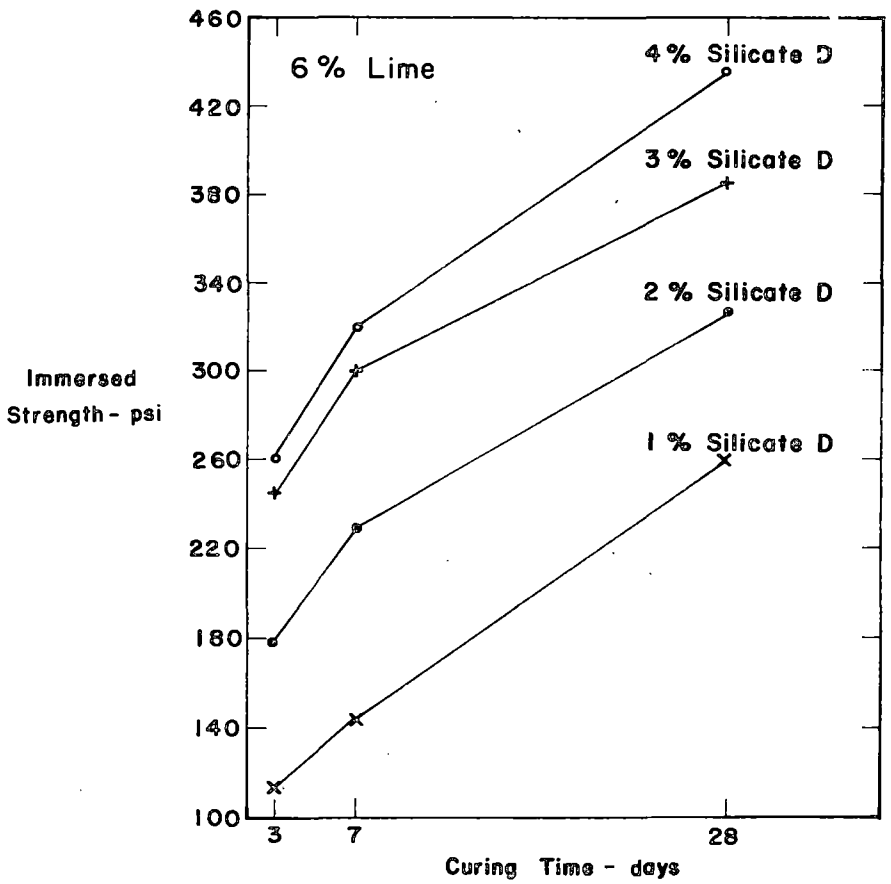


**Fig. 5. Effect of curing time on 1 day immersed unconfined compressive strength of Kansan till stabilized with 6% calcitic hydrated lime and various percentages of silicate D.**

*Discussion.* The effect of curing time on 1 day immersed unconfined compressive strength of Kansan till stabilized with 6% calcitic hydrated

lime and varying percentages of silicate D is shown (figure 5). All four mixtures continued to gain strength throughout the entire period investigated. The average rate of strength increase for all mixtures was greatest in the period 3 to 7 days. The more silicate D added, the higher the average rate of strength gain was for this period. For the period 7 to 28 days, the average rate of strength gain for the 1, 2 and 3% silicate D mixtures was approximately the same. The addition of 4% silicate D, however, increased the average rate of strength gain for this period. Further investigation of the reaction products formed by these combinations of soil, lime, and silicate D are needed before an explanation for this can be given.

**Resistance to Freeze-Thaw Action**

The four mixtures investigated in the curing time study were used for a study of their resistance to freezing. The four mixtures, after curing for seven days were subjected to fourteen cycles of freezing and thawing and then tested for unconfined compressive strength (table VII).

TABLE VII. RESULTS OF MODIFIED BRITISH FREEZE-THAW TESTS OF KANSAN TILL STABILIZED WITH 6% CALCITIC HYDRATED LIME AND DIFFERENT PERCENTAGES OF SILICATE D.

| Silicate D content, % | $p_c$*, psi | $p_f$†, psi | $R_f$‡, % |
|---|---|---|---|
| 1 | 195 | 6 | 5 |
| 2 | 281 | 116 | 40 |
| 3 | 307 | 179 | 60 |
| 4 | 429 | 218 | 50 |

*Unconfined compressive strength of immersed specimen.
†Unconfined compressive strength of freeze-thaw specimen.
‡Index of the resistance to the effect of freezing.

*Discussion.* Tentative criteria for durable soil cement from the data obtained in the modified British freeze-thaw test have been adopted at Iowa State University. The first criterion is the $R_f$ value (the ratio of the unconfined strength of the immersed specimens to the unconfined compressive strength of the freeze-thaw specimen expressed to the nearest 5%) which must equal or exceed 80% for soil cement. The second criterion is that the unconfined compressive strength of both the control specimen and the freeze-thaw specimen must equal or exceed 250 psi[4].

There is no reason to dispute the second criterion in the evaluation of the durability of the lime and silicate D stabilized soil. However, the validity

of the first criterion (the $R_f$ value) as applied to lime and silicate D stabilized soil is questionable.

It has been found that after an initial moist curing of one day specimens of lime and sodium silicate stabilized soil immersed in distilled water for seven days yielded higher unconfined compressive strengths than specimens cured for seven days in the moist room and immersed for one day before testing[6]. This coupled with the probability that the freezing inhibits the gain of strength of the specimen during the freeze-thaw tests, seems to indicate than an $R_f$ value of 80% may not be a valid criterion for freeze-thaw resistance of this type of stabilized soil. Further investigation of this criterion is needed before any final evaluation of results of the freeze-thaw tests of lime and sodium silicate stabilized soil can be made.

None of the mixtures tested were able to pass the criterion of an unconfined compressive strength equal to or in excess of 250 psi after fourteen freeze-thaw cycles (table VII). However, the addition of silicate D increased the strength of the specimens at the end of fourteen cycles of freezing and thawing until the combination of 6% calcitic hydrated lime and 4% silicate D came very close to meeting the criterion. The iso-strength contour chart (figure 6) indicates that a combination of 7% calcitic hydrated lime and 4% silicate D would produce a stabilized soil with greater strength than the combination of 6% lime and 4% silicate D. It is probable then that Kansan till can be made resistant to freeze-thaw action by treatment with a combination of calcitic hydrated lime and silicate D. The exact combination of these materials needed is subject to further investigation.

## SUMMARY AND CONCLUSIONS

The investigation reported herein is divided into four parts. The first part reports, discusses, and offers an explanation for the effect of ageing time on dry density on seven day cured, one day immersed unconfined compressive strength, and on optimum molding moisture content for maximum dry density and maximum strength of Kansan till stabilized with 6% calcitic hydrated lime and five different powdered sodium silicates. The amounts of sodium silicates used were adjusted so that the anhydrous portion of each was equal in weight to the anhydrous portion of 6% sodium metasilicate pentahydrate.

The second part reports the selection of the optimum combination of lime and sodium sesquisilicate pentahydrate, the sodium silicate whose strength was least affected by ageing time. The effect of various combinations of lime and this sodium silicate on the optimum molding moisture content for maximum dry density and maximum strength is also commented on.

The third part reports and discusses the effect of curing time on strength of various combinations of sodium sesquisilicate and calcitic hydrated lime.

The fourth part reports and discusses the resistance of the soil stabilized with 6% calcitic lime and various percentages of sodium sesquisilicate to freeze-thaw action.

1. Kansan till treated with 6% calcitic hydrated lime and any one of the five powdered sodium silicates investigated displays the following characteristics:

a. As ageing time increases, dry density decreases at a rate depending on the type of sodium silicate.

b. Ageing time affects optimum molding moisture content both for maximum dry density and for maximum strength, but the effect is not the same.

c. As ageing time increases, strength decreases. The amount of this decrease depends on the molding moisture content as well as the type of sodium silicate.

d. Strength is not correlated with dry density.

2. Sodium sesquisilicate pentahydrate (silicate D) when mixed with 6% calcitic hydrated lime and Kansan till produces a stabilized soil whose strength and dry density at time of molding are less affected by ageing time than any of the other sodium silicates studied.

3. Of the combinations of lime and silicate D studied, the optimum mix design for maximum strength was 6% lime plus 4% silicate D.

4. For the mixtures of lime and silicate D studied, the rate of strength increase with curing time is greater in the period 3 to 7 days than in the period 7 to 28 days.

5. In the period 3 to 7 days the rate of strength increase with curing time increases as the silicate D content increases.

6. Three day cured, one day immersed unconfined compressive strengths of over 250 psi, can be achieved by mixtures of calcitic hydrated lime and silicate D.

7. Kansan till stabilized with 6% calcitic hydrated lime and amounts of between 2 and 4% silicate D probably would perform satisfactorily as a pavement base course in an area with a mild climate.

8. In field construction using lime and sodium silicate stabilized soil, strict controls over moisture content and elapsed time between final mixing and compaction will be necessary to obtain the desired strength.

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